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Laboratory Notes.

IV.

Laboratory Notes.

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Analysis of Malt Extract. 1888.

Thomsens Malt Extract, made from barley, and very pure.

Maltose	62.72 %
Dextrine	10.33 "
Albuminates	1.66 "
Free acid (Calculated as Lactic)	.72 "
Alcohol	1.23 "
Ash	1.43 "
Water	22.20 "
Total	100.29

Process of analysis. -

Maltose (Malt sugar) - Determine with Fehling's solution. Place 10. c.c. Fs. sol. in a flask, dilute with 2 to 4 volumes of dist. water and heat to boiling. Then add, from a graduated burette, a 2 % solution of malt extract, continuing the boiling, and shaking the flask. Add malt solution

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until the last shade of green in Fs. Sol. has just disappeared. The faintest shade of green can be detected by holding the solution up to the light.- If desired, the solution can be tested for copper by filtering a few drops, adding acetic acid and then Ferrocyanide of Potas. If copper still remains in solution, a brownish color will be produced.

10. c.c. Fs. Sol. is reduced by 0.074 gram Maltose. From these data results are calculated.

Dextrine. The dextrine is first converted into Glucose by boiling 4 grams of malt extract dissolved in 200. c.c. water on water bath with 20 c.c. HCl of 1.125 Sp. gr. for several hours, replacing the evaporated water. Then determine total Glucose with Fehling's solution. Maltose is converted into an equal amount of Glucose by boiling with

$N.Cl$; hence the % of Maltose found must be deducted from the total % of Glucose. Glucose remaining will represent the dextrine in Malt Extract.

100. parts Glucose = 91.67 pts. Dextrine.

10. c.c. Fehling's sol. = 0.05 grains Glucose.

Albuminates. To 100. c.c. of a 10 % solution of Malt Extract add 100. c.c. of cold saturated solution of Picric acid, and digest at about digestive heat (100° F.) for ½ hour. This will ppte. albuminates completely. Collect ppt. on Tared filter paper, wash with water, dry at 100° C. and weigh. - Albuminates = ½ of weight obtained.

Free Acid - Calculated as Lactic acid - ($C_3H_6O_3$ - 90.) Dissolve a known quantity of Malt Extract in dist. water and titrate with Normal Ammonia Solution.

1000. c.c. Normal Sol. = 90. grams Lactic acid.

1. " " " " = .090 " " "

Alcohol. Place 500.c.c. of a 10% solution of Malt Extract into a retort, retort should not be more than half filled. Distill until alcohol has all passed over. Weigh distillate in pyknometer or determine sp. gr. with hydrometer, and find % of alcohol by referring to Table of Alcoholimetry.

Water. — Place 10 grams of freshly ignited quartz sand in a platinum dish and weigh. Add ~~to~~ 10.c.c. of a 10% Sol. (= 1.gr.) of Malt Extract and evaporate, first on water bath and then in hot air bath at 110° C. until weight is constant.

Difference = water.

Ash. Ignite last residue, until all organic matter has been burned off. Residue = Ash.

Glycerine. - Glycerine is often added to Malt Extracts, and it can hardly be called an adulteration. It can be determined by extracting

with a mixture of 1 vol. ether and 2 vols. Absolute alcohol - Evaporating to dryness - again extracting residue with same mixture and again evaporating clear solution. Residue = Glycerine.

References.

Maltose - See p. 591 Fresenius' Quantitative Analyse - Vol. II. German Edition.

Diastase. See p. 460 Fresenius Zeitschrift - Vol. 21.

Albuminates. See p. 125 Fresenius Zeitschrift - Vol. 16.

Glycerine - See p. 341 Hager's Chemische Technologie.

Diastase. 100 parts of good Malt Extract should convert about 10 pts. of Starch into sugar.

Of Thomsen's Extract - 100 pts. converted 9.1 pts. Starch into sugar.

Diastase is determined as follows: Dissolve 0.5 gram Starch in 500 c.c. boiling water, and divide into 5 equal parts. Add to this aliquot parts

8 c.c., 11. c.c., 14. c.c., 17 c.c. and 20 c.c. respectively of a 10% solution of Malt Extract. Digest at a temperature of 35° to 40° C. for three hours, shaking or stirring from time to time. At the end of 3 hrs. begin with the one to which 20 c.c. N.E. sol. has been added and test for Starch with an Iodine solution, until the one is reached which gives a faint blue. This one represents the amount of Starch converted into Sugar by the amount of Malt Extract added to it.

Remarks.

Malt Extract is very frequently adulterated with glucose Syrup. It is difficult directly to determine this directly. If a Malt Extract is largely adulterated with glucose, then the amount of albuminates is usually very small, and a solution of the Extract will yield but a comparatively small milkiness with Picric

acid solution.

Again – in the presence of Glucose, the total of the percentages of the constituents will be greater than 100 – for Glucose would naturally be estimated as maltose and it takes but .05 gr. glucose to reduce 10 cc. Fehling's solution, while it requires 0.074 gr. Maltose to reduce 10 cc.

Good, pure Maltose should yield nearly a clear solution with water – with no large amount of insoluble matter.

Fehling's Solution.

A solution of 34.65 grams of pure copper sulphate in 200. c.c. water, is mixed with a solution of 100 to 150 grams of Rochelle salt in 500 c.c. of a 10% solution of NaHO, (1.14 Sp. gr.) — and dilute to 1 Litre.

10 c.c. of this sol. = .05 gram glucose.
10. " " " " = .074 " Maltose
100 pts. Glucose = 91.67 pts. Dextrine,
180 " Glucose = 162. " Starch.

For mode of using see p. 3.

Mayer's Solution for Alkaloids.

The 1/10 solution is prepared as follows:-

Dissolve 49.8 grams of Potassium Iodide in water and pour into a solution of 13.546 grams of Mercuric Chloride ($HgCl_2$); dilute to 1 Litre.

1 c.c. of this solution will precipitate of-

Aconitin	.02680 Gram.
Atropia	.01450 "
Brucia	.02330 "
Quinina	.01080 "
Quinidina	.01080 "
Cinchonina	.01020 "
Cotnina	.00416 "
Morphina	.02000 "
Narcotina	.02130 "
Nicotina	.00405 "
Strychnina	.01670 "
Veratrin	.02690 "

Method for using Mayer's sol. in quantitive determination of Alkaloids.

The solution of the alkaloid should be that of a salt, and should be neutral or slightly acid. Add a small quantity of Mayer's sol. from a burette, stir and allow ppt. to settle. Then by adding another drop of M's. Sol. it can easily be seen if more ppte. is formed. Towards the end of the operation, it is well to take a drop or two of the clear supernatant fluid, place it on a dark glass and add a drop of M's. sol. If precipitation was not complete, a ppte. will at once form, and the operation will have to be continued.

Solution Molybdate of Ammonium for Precipitation of Phosphoric Acid.

Dissolve 150. grams of Molybdate of Ammonium, C.P. in 1000 c.c. Water. Pour this solution into 1000. c.c. (1 L.) of pure HNO_3, One. 100 c.c. of this solution is the equivalent of 0.1 gram of Phosphoric acid.

Some Fire Extinguisher Solutions analyzed. —

1. - A solution of $CaCl_2$ in H_2O.
2. A solution of sulphite of ammonium in water.
3. A solution of common salt with a small amount of $CaCl_2$, $CaSO_4$ in water - also a small quantity of free NH_3.
4. Solution of $NaCl$, $CaCl$, NH_4Cl, $CaSO_4$ + free NH_3 in water.

Copper ore - Scheme for determination of copper.

Powder ore finely and take samples from .5 to 2. grams, varying with the richness of ore. Place in a porcelain dish and add in the order named 10 c.c. HCl, 5 c.c. HNO_3 & 10 c.c. H_2SO_4 all concentrated. Evaporate first on water bath and then over the flame direct, until dense white fumes of sulphuric acid come off in profusion. Allow to cool and dilute with water, filter. Put filtrate, or aliquot portion thereof into a platinum dish and precipitate metallic copper either with battery or with iron. -

With battery. attach copper wire to each pole of battery - twist the end of wire attached to Carbon pole into a spiral and upon this spiral set the platinum dish containing the solution. To the end of the wire attached

to the Zinc pole fit a thin strip of Platinum foil; this foil immerse into the Cu solution in the dish. The Cu will separate on the sides of the platinum dish. When precipitation is complete, which can be determined by testing a few drops with H_2S, pour out the liquid wash first with water, then twice with alcohol, then dry holding dish in hand and passing it back and forth over a Bunsen burner flame.

With Iron. Put into platinum dish containing Cu solution, 3 or 4 bright iron nails, leaning them against side of vessel, and allow to stand for some hours. When precipitation is complete, treat Cu as above.

Determination of Nitrogen in Fertilizers.

Fertilizers being mixtures of bones, manure etc. contain from 4. to 7 or 8 % NH_3.

Blood fertilizers as high as 14. to 18. % of NH_3.

Take about 1. gram sample, and determine N by ordinary combustion process, passing NH_3 into Normal H_2SO_4 and titrating with Normal NH_4HO. (See Fresenius)

Tin (Sn), Wet assay.-

Take sample from 1. to 5. grams, varying with richness of ore. Pulverize very finely. Mix ore with 3 parts of pure sulphur and 3 parts of dry Carbonate of Soda. Fuse the mixture in a well covered porcelain crucible over a Bunsen burner for 15 to 20 minutes. Allow to cool and macerate with water; this will dissolve Na_2S formed and hence also the SnS_2 formed. Filter - if there is still undecomposed residue, it should be again fused as above. In the filtrate the Tin is precipitated as Sulphide ~~with~~ by acidifying with HCl. Allow to settle and filter and wash with water. The Sulphide of Tin is then washed into a beaker and dissolved in HCl containing Bromine in solution, warming somewhat. When completely dissolved, add Aqua Ammon. slowly, until a permanent white precip-

state appears, then add HCl until the ppte. is just redissolved, leaving the solution slightly acid. To this solution add a saturated solution of NH_4NO_3 which completely precipitates the Sn as hydroxide. Allow to stand over night. Draw off the clear fluid filter, and wash ppte. 3 times by decantation, then transfer it to filter, wash dry, ignite and weigh as SnO_2. (See Fresenius' Quantitative Analyse, original German edition, Vol. II, p. 545.)

If the ore contains Tungstic acid (Wolfram) this will be weighed with the SnO_2. In such a case after weighing the two, they should be ignited, repeatedly with NH_4Cl, by which the SnO_2 will be completely volatilized, leaving the Tungstic acid, which can then be weighed.

Tin – Fire Assay. (SnO_2)

Sample of the powdered ore is mixed with powdered charcoal and a small amount of Bi-carb. of Soda. Covered with salt, and charcoal; then submit to a high heat in furnace for 30 to 45 minutes. Button Tin will collect at bottom.

If button is not pure, Tin can be determined in it by boiling in HNO_3 (sp. gr. 1.3) in glass flask; as soon as reaction has ceased, transfer to porcelain dish, evaporate nearly to dryness, dilute with H_2O, filter and wash. Ignite and weigh residue as SnO_2.

Tin – Scheme for, II.

Take sample of 1 to 5 grams. Boil with Nitro-Hydrochloric acid – then transfer to porcelain dish, & evaporate to dryness. Digest with a little acid and water, filter – Residue = SiO_2 & SnO_2. Dry and ignite

Fuse with dry ppt for ten times its weight of KHO in a Silver crucible. KHO placed in crucible with its own weight of water and above residue added. Mass is then evaporated to dryness and heated for ½ hour until fusion is complete. Dissolve fused mass in water and acidify with HCl; filter and bring to dryness. Digest with HCl & H2O & filter. Solution = Sio2; residue = Li2O. Treat this HCl solution as given at bottom of p. 23 & top of p. 24.

Examination of Samples of Licorice Powder for Belladonna (Atropia), Henbane (Hyosciamin) and Stramonium (Daturin).

Sample ~~made~~ shaken with KHO solution and Chloroform. Chloroform extract drawn off and filtered. A portion of filtrate shaken with dilute H_2SO_4 (very dilute) H_2SO_4 drawn off and tested with Mayer's solution, $PtCl_4$ and Iuric Chloride.

The balance of Chloroform solution evaporated to dryness on water bath extracted with dil. H_2SO_4, filtered and filtrate tested for Alkaloids with Mayer's solution, Platinic and Iuric Chloride.

A sample of Senna was tested in the same way.

Hydraulic Cement.

Analysis of rock to see if it is of value for making hydraulic cement.

Boil a sample with HCl, filter dry residue ignite and weigh. – Report this as insoluble matter – or if desirable – fuse and make analysis of insoluble matter. SiO_2, Al_2O_3, Fe_2O_3, CaO – MgO, Na + K. –

Analyze the portion dissolved in HCl separately. Fe_2O_3, Al_2O_3, CaO + MgO. –

$CO_2 + H_2O$ by difference.

Compare with Hager and other authorities.

Milk. Examination to determine whether or not diluted with water. Sample should be perfectly fresh and sweet.

Take 10 to 15 grams, mix in platinum dish (tared) with either 10 grams to 15 grams of freshly ignited quartz sand or Oxide of ~~Iron~~ (Hydroxide) or with 5 to 10 grams of dry pulverized Charcoal. Evaporate on water bath and dry in hot air bath at 110° C.— Weigh residue.—

Pure milk should contain 10 or 11 to 14 or 15 o/o of total solids.—

Paint-Red. Analysis of a number of samples.—

1.	Red Lead (Pb_3O_4)	74.70 o/o.
	Alumina (Al_2O_3)	12.95 "
	Volatile Matter —	6.62 "
	Impurities – Fe, Ca Mg, SiO_2, Na + K	5.73 "
		100.00
2.	Red Lead —	93.87 o/o
	Volatile Mat. (H_2O etc.)	3.08 "
	Impurities – Fe, Al, Ca etc. by difference	3.10 "
		100.00 "
3.	Red Lead —	71.06 o/o
	Alumina —	20.24 "
	Volatile Matter —	3.82 "
	Silica —	2.42 "
	Impurities, Ca, Mg, Fe, etc. by difference	2.46 "
		100.00

Red Paint – Course of analysis.
Take ½ to 1 gram. Boil in flask with dilute HNO_3 – this will dissolve portion of Oxide of Lead, leaving a dark brown oxide undissolved. Add alcohol and again heat – carfully this time, else the action may become so energetic as to force some of liquid out of top of flask. If there is an undissolved precipitate remaining, filter and wash – Call residue "A."

Take all or aliquot portion of filtrate and precipitate Pb as $PbSO_4$ with dilute H_2SO_4, adding two volumes of alcohol. – Allow ppte. to subside, filter, wash with alcohol (2 vol. Alcohol + 1 water) dry. Ignite $PbSO_4$ in usual way.

Test filtrate from $PbSO_4$ for Hg, Ba, Ca &c

Residue "A" is ignited (and weigh

ed) and fused with Na_2CO_3. Macerate with water, test for SO_3 and Ba - if neither of these is present, acidify with HCl - If SiO_2 is present, Evaporate to dryness and determine SiO_2 in usual way.

Opte. Al_2O_3 with NH_4HO and determine in usual way. -

Volatile Matter (Water etc) Ignite sample (1-gram) in porcelain crucible and weigh. From difference there should be deducted the molecule of Oxygen which is driven off from Pb_3O_4. Difference = volatile matter.

Leather Dressing (Polish) for Buggy tops, Harness etc.

To make 10. gallons dressing: -

Water	9. gal. 3½ pts.
Aqua Ammon. 0.9 Sp. Gr.	1⅓ pt.
Extract Logwood (Solid)	2 lbs. 1⅓ oz.
Bichromate of Potas.	2. oz.
Borax — — — — —	2. lbs. 12½ oz.
Shellack ———	6. " 7½ "
Oil Sassafras ——	3⅓ "
Coloplony	1 " 10. "
Soda (dry) ———	13. "

Last two boiled separately in 3 pints of water until dissolved and added.

Analysis of Dressing something like above. Total Residue. Evaporate to dryness on water bath and in hot air bath at 110°C. -

~~Bichromate~~ Borax. - According to authorities found that about 4 to 5 parts Borax required to dissolve 10 parts of Shellack.

Shellack. By precipitation with Ac_2. Filter on sand filter, dry and weigh.—

Logwood and Bi-chromate of Potas. From total solids subtract sum of Shellack and borax — difference = Logwood and Bi-chromate. According to authorities 15. parts bi-chromate should be added to about 250. parts Logwood extract.—

NH_4HO, — add excess of Normal H_2SO_4 and titrate back with Normal NH_4HO.—

Oil of Wintergreen. By distillation.

Water — By difference.

Borax can be determined by precipitating Shellack with excess of Ac_2 — destroying Logwood with Cl and evaporating to dryness & igniting. B_2O_3 will all be expelled and Borax can be estimated from Mass formed.

Antimony — In Ores. Determination
Sample .3 to 1. gram. Treat with
Nitro-Hydrochloric acid, evaporate
to dryness in porcelain dish.
Dissolve in HCl and water, add
Tartaric acid, filter, precipitate
Sb with H_2S, allow to subside, fil-
ter on tared filter paper, dry and
weigh as Sb_2S_3. —

In impure ores, it will be
necessary to take further precau-
tions, purifying the Sb_2S_3.

Borax — Assay. Titrate with Normal H_2SO_4. —

As sample take 5. grams Borax.

1. c.c. Normal H_2SO_4 = .191 gram of crystalized Borax.

Sample of Medicine.

The following is a copy of Prescription:

" Acid Carbolic	.5	grams.
Tinct. Gelsemin	15.	"
Glycerine	15.	"
Ext. Cascara Sagrada	15.	"
Tr. Opii Camphorated	30.	"
Aqua, q.s.	120.	"

Teaspoonful before each meal."

In the above were determined the total alkaloids (Morphia and Gelsemin), with a view of ascertaining whether or not the were in excess of what the prescription called for.

20.06 grams of the medicine were made strongly alkaline with KHO and then thoroughly shaken and washed with Chloroform. The Chloroform Extract was evaporated to dryness on water bath. The alkaloids in the residue were dissolved in dilute H_2SO_4 and determined vol-

numerically with Mayer's solution. 1/100 Mayer's solution was added until a few drops of the Extract (H_2SO_4), placed on a dark glass plate no longer gave a ppt. with a drop of Mayer's solution.

1/100 Mayer's Sol. added = 2. cc.
1 cc 1/10 " " = .02 gram Morphia
2 " 1/100 " " = .004 " "

I was unable to find, in the literature on the subject, just what proportion of Gelsemin is precipitated by Mayer's solution. But, as doubtlessly the amount of Gelsemin precipitated cannot vary greatly from the amount of Morphia so precipitated, they were estimated together as Morphia.

20.06 : .004 :: 100 : x = .0199 % Alkaloids in Medicine, found by actual determination.

Now to estimate the amount of Alkaloids the medicine should con-

Tain according to prescription: —

Tinc. of Opii Camph. contains 4 o/o Opium, Thus of the 30 grams of Tr. Opii Camph. contained in the 120 grams of medicine 0.120 gram = Opium. Now the abvrage amount of Morphia in Opium = 11. o/o. — 11 o/o of .120 = .0132 gram Morphia in 120. grams of medicine. 120.: .0132 :: 100 : 4 = .011 o/o Morphia.

Again: —

120. grams Medicine contain 15. grams Tinct. Gelsemin.

100. grams Tinct. Gelsemium contain 15. grams Gelsemium.

100 : 15 :: 15 : 4 = 2.25 Grams Gelsemium in 15 grams Tinct. or 120 grams of above medicine.

Gelsemium contains .5 o/o Gelsemin (alkaloid).

0.5 o/o of 2.25 = .01125 = Gelsemin in 15 grams Tinct. or 120 grams medicine.

120 : .01125 :: 100 : X = .0093 %
Gelsemin (alkaloid).

Results :—

Gelsemin estimated from prescrip. = .0093 %
Morphia " " " " .0110 "
Total Alkaloids " " " " .0203 "
" " by actual determin. .0199 "

Hence amount of alkaloids in medicine is not in excess of the amount called for in the prescription.

Manganese. Wet assay in Or.
Sample - 1/2 to 2. grams.
Dissolve in excess of HCl. Ppte. Fe
+ Al with NH_4OH after adding NH_4Cl.
Boil + filter. Filtrate (a) preserved.
Dissolve ppte. of Al + Fe and again
precipitate as above. Filter - fil-
trate added to a. Place in a flask,
and precipitate Mn with $(NH_4)_2S$,
and fill to neck with water. Allow
to stand over night. Filter and
wash, first by decantation, then on
filter, with water containing $(NH_4)_2S$
+ NH_4Cl. Dissolve ppte. in HCl,
heat until odor of $(NH_4)_2S$ has
passed off, ppte. Mn. with Na_2CO_3,
heat, filter and wash very thoroughly.
Dry, ignite and weigh as Mn_3O_4.

Kaolin.

Analysis.

SiO_2	65.654	o/o.
Al_2O_3	17.300	"
CaO	3.178	"
MgO	1.030	"
Fe_2O_3	1.067	"
Na_2O	.803	"
H_2O	11.018	"
	100.000	

Kaolin.

Analysis.

SiO_2	43.090	o/o,
Al_2O_3	38.780	"
CaO	1.180	"
MgO	.194	"
Fe_2O_3	.577	"
$Na_2O + K_2O$	1.485	"
H_2O	14.694	"
	100.000	

Brick. (Supposed Fire Brick)
analysis.

SiO_2	61.59	o/o
Al_2O_3	32.84	"
Fe_2O_3	3.44	"
CaO	1.24	"
MgO	.36	"
Na_2O	.43	
K_2O	.10	
	100.00	

Limestone. (Coarse, impure quality)
analysis.-

SiO_2	10.95	o/o
Al_2O_3	1.63	"
Fe_2O_3	.73	"
CaO	29.68	"
MgO	15.54	"
Alkalies	.07	"
CO_2	40.62	"
	99.22	"

Clay - Partial analysis.

Alkalies	——	2.480 o/o
MgO	——	.176 "
CaO	——	2.996 "
Fe_2O_3	——	8.030 "
Cl and SO_3	——	Traces.

Clay - Partial analysis.

Alkalies	——	1.450 o/o
MgO	——	.450 "
CaO	——	1.618 "
Fe_2O_3	——	5.260 "
Cl + SO_3	——	Traces.

Clay - Analysis.

SiO_2	——	72.67 o/o.
Al_2O_3	——	13.84 "
Fe_2O_3	——	3.35 "
CaO	——	1.31 "
MgO	——	Trace "
Na_2O	——	1.69 "
K_2O	——	Trace "

Clay - analysis.

$Si\,O_2$ ——	56.15 %
Al_2O_3 ——	25.51 "
Fe_2O_3 ——	8.56 "
CaO ——	1.20 "
MgO ——	.50 "
Alkalies —	7.08 "
Water	1.00.
	100.00 —

Granite, Sandstone, Felspar,
Clay, Kaolin, Brick, Limestone etc.

Course of analysis.

Samples of clay, Kaolin, Brick or Sandstone are finely powdered.

Sample = 1. gram. -

Fuse with dry Na_2CO_3 and a small quantity of KNO_3, in Pt. crucible. Boil, digest with water, dissolve in HCl, evaporate to dryness. -

$Si O_2$. Separate in usual way, by moistening residue with HCl adding water, digesting and filtering. Sometimes a second fusion is necessary, usually when Al_2O_3 is high.

Divide filtrate from $Si O_2$ into two equal portions, each representing 1/2 gram of sample. Precipitate in each, separately the $Al_2O_3 + Fe_2O_3$ with NH_4HO.

Al_2O_3. Filter both; the one ignite and weigh $Al_2O_3 + Fe_2O_3$ together.

Fe_2O_3. The other dissolve in Dil. H_2SO_4, reduce Fe with Zn and titrate with Permanganate of Potas. Sol.

Deduct the Fe_2O_3 thus found from sum of $Al_2O_3 + Fe_2O_3$ and balance $= Al_2O_3$.

The two filtrates received after precipitating $Fe_2O_3 + Al_2O_3$ are exactly alike — call them "A." + "B," and each represents ½ gram sample.-

In "A" precipitate CaO with Ammonium Oxalate - boil, allow to stand for several hours - filter. Dry ~~ppt~~. and ignite over blast lamp to reduce completely to CaO.

In filtrate from CaO precipitate MgO_4 by adding Di-Sodic Hydric Phosphate. Allow to stand over night.- Filter, ignite and weigh as $Mg_2P_2O_7$. Estimate as MgO.-

~~In "B," precipitate all the CaO + MgO by adding $(NH_4)_2O_3$ in excess, then a small amount of $(NH_4)_2C_2O_4$ boiling and allowing to stand over night. Filter; in filtrate determine Alkalis.~~

in Silica-stone 5/12

$Na_2O + K_2O$. Acidify filtrate with HCl, boil in beaker covered with watch-glass until CO_2 has passed off, evaporate first in beaker if bulk is large, then transfer to platinum dish, evaporate to dryness on water bath then carefully ignite until all $NH_4Cl + Cl$ has been volatilized, and weigh residue as $NaCl + KCl$.

Separate these two by dissolving in smallest possible quantity of water, add Platinic chloride, evaporate almost to dryness, add alcohol of about 85%, filter on hard filter, wash with alcohol of 85%, dry and weigh as K_2PtCl_6. Estimate as K_2O.

Na_2O. $NaCl$ = difference between $NaCl + KCl$ and KCl. Estimate as Na_2O.

In analysis of Limestone fusion is not necessary, but simple solution in HCl. $Na + K$ can be directly determined in filtrate from $Al_2O_3 + Fe_2O_3$. — CO_2 is determined in CO_2 ap-

paratus.

Na + K in Clay, Brick, Kaolin, Sandstone

Sample finely powdered — 1. to 4. grams. Place in platinum dish and add mixture of equal parts of conc. H_2SO_4 & water poured over it; evaporated to dryness, finally over lamp direct. This operation is repeated. Moisten with HCl add water & digest. Filter and wash; ppte. Fe al with NH_4HO, filter; ppte. CaO & MgO with $(NH_4)_2CO_3$ and a little $(NH_4)_2C_2O_4$, boil - allow to stand over night. Filter and proceed as given on p. 51. Weight as sulphates or if K & Na is to be separated convert into chlorides (see Fresenius) and weigh as such.

Lampa Building Stone.

Analysis and Examination.

SiO_2 ——— 59,31 o/o

Al_2O_3 ——— 22.39 "

Fe_2O_3 ——— 3,57 "

CaO ——— 1,88 "

MgO ——— ,57 "

Alkalies ——— 12.33 "

Cold HCl (conc) poured over the somewhat coarsely powdered rock — of — Al_2O_3 ——— 5,84 o/o

Fe_2O_3 ——— 2,85 "

Water absorbed after immersion for 24 hours = 0,444 o/o of its weight.

Specific gravity — 2,481

Rock contains no H_2S. —

Concentrated solution of $MgSO_4$ had no disintegrating effect on it.

Hydrocyanic Acid (HCy or HCN). 2% Solution

Preparation of. —

To make one litre use —

175. grams coarsely powdered $K_4Fe(CN)_6$.

125. " Conc. H_2SO_4

250. to 350. cc. Water.

Place into glass retort and distill on oil bath until HCy ceases to come off. Temperature of oil bath 115° to 130° C

Cease distillation when solution in retort begins to bump excessively.

In diluting to 1000. cc. it is well to use 50% alcohol. —

Solution may be tested by titration with $\frac{1}{10}$ $AgNO_3$ solution.

Acid Phosphate,
Examination of.
Solubility in water. Digest 2 grams with water, filter on Fand filter dry and weigh residue.
Acidity. Place weighed portion (sample phosphate) in CO_2 apparatus, and strong solution Na_2CO_3 in top of apparatus. Weigh. Bring the two together, allow to effervesce and finally draw air through to expel CO_2. Weigh - Diff. = CO_2 expelled by phosph. -

Analysis. -
Soluble portion = 52.785 o/o. -
2 2.28 parts Na_2CO_3 neutralize 100 pts. phosphate.
35.31 parts $NaHCO_3$ neutralize 100 pts. phosphate.

Embalming Fluids.

Analysis. —

NaCl	.0702 o/o
$CaSO_4$	.0240 "
Oil Citronella	.0762 "
Boric acid (B_2O_3)	.1098 "
Water	99.7598 "

Another sample was an alcoholic solution of Boric acid.

Boric acid — 0,324 o/o.

Oil citronella was estimated by adding Oil Citronella & water until approximately the same strength (odor) was obtained.

Boric acid by difference.

Carriage-Top Dressing.

Analysis.

Asphaltum	20.627	%
Colophony (Rosin)	15.000	"
Vaseline	2.920	"
Oil Turpentine	61.453	"
	100.000.	

Course of analysis. - Digest 20 grams with Ether - filter on tared filter, dry residue and weigh. This equals a portion (A) of Asphaltum insoluble in Ether.

To Ether solution add alcohol and evaporate Ether at low temperature. Again filter and weigh residue. This = portion (B) of Asphaltum soluble in Ether and Vaseline.

Vaseline. To determine approximately the proportion of vaseline in B, varying proportions of Vaseline and Asphaltum were mixed and fused together until a mixture the consistency

of B was obtained. The amount (C) of vaseline used in this mixture ~~take~~ as amount vaseline in Dressing. –

Asphaltum,

B – C + a = Total asphaltum.

Rosin. – To alcoholic solution add water and evaporate to dryness. Residue = Rosin. (?)

Oil Turpentine by difference.

On Experimenting and making up a sample, it was found that the following proportions gave good results: –

Purified Asphalt.	—	25. parts.
Rosin (Colophony)	—	10. "
Common Beeswax	—	3. "
Vaseline	—	3. "
Oil Turpentine		59. "

On further Experimenting, with a view of finding some way of making

the dressing dry more rapidly, it was found that Litharge and Sugar of Lead gave unsatisfactory results.

Japan Drier Laurier was found to work very well.

Use following proportions:-

Asphaltum	18.2	parts.
Rosin	7.3	"
Beeswax	2.2	"
Vaseline	2.2	"
Oil Turpentine	43.0	"
Japan Drier	27.0	"

Iodine and Bromine — Determination of — in water in presence of — I, Br & Cl. —

(See Fresenius' Zeitschrift, Vol. 18, page 478).

Iodine. Evaporate 500 c.c. water to small bulk. Acidify with H_2SO_4, add acid sulphite (hyposulphite) of Soda Sol. and $Cu\,SO_4$ solution. Allow to stand for some time; this precipitate I as "Cu_2I_2". Filter on tared filter, dry and weigh. —

Bromine. Boil filtrate from Cu_2I_2 with excess of H_2SO_4 until sulphurous acid is all expelled. Transfer to a glass flask, which is connected by a tightly fitting (rubber stopper) glass tube with bulbs containing acid sulphite of Soda (hyposulphite) solution. The bulbs are immersed in cold water. Then add to contents in flask a mixture of solution of Bi-chromate of Potass. and Sulphuric acid. Then

boil until all the Bromine has been distilled over into bulbs. Empty bulbs into beaker, add excess of HNO_3 and precipitate Br with $AgNO_3$; filter, dry, ignite & weigh. Care must be taken to add a sufficient quantity of HNO_3 to redissolve all the Sulphite of Silver that may be precipitated.

Soap. Analysis.

Fatty acids	66.76 %
Water	25.54 "
Alkali (combined)	7.52 "
" (free in excess)	.15 "

Course of analysis.

Fatty acids. Take 10. grams of soap, dissolve in water, separate Fatty acids by adding excess of HCl, ~~draw~~ allow to cool, draw off clear liquid, wash, dry and weigh.

Water. Take 5. grams finely sliced soap, place into platinum dish dry first on water bath and finaly in hot air bath between 100° & 110° C, until weight is constant. Water = difference.

Alkali, Total (Na_2O). Take 2 grams soap, dissolve in water and add excess of Normal HCl (or H_2SO_4) and titrate back with Normal NH_4HO. Difference represents Na_2O.

Alkali in Excess (free). Dissolve 5 or more grams soap in water, boil with excess of NaCl, allow to cool and draw off clear solution. NaCl must be C.P. Titrate clear solution with Normal HCl or H_2SO_4. Acid required represents Na_2O free. — Deduct free Na_2O from total Na_2O to get combined Na_2O. Sup. 93

Titanium (Titanic acid)
Determination in Iron Ore,
Ilmenite.

Take 1 to 5 grams. Fuse with Na_2CO_3; dissolve fused mass in excess of H_2SO_4, cautiously evaporate to dryness and gradually raise the heat until the bisulphate of soda formed passes into fusion at a low heat. Allow to cool and add cold water. Let it stand for several hours, until thoroughly softened and dissolved; dilute to 500. c.c. filter to separate SiO_2; to the filtrate add sulphurous acid, and heat to boiling for an hour or more, adding more sulphurous acid from time to time. The Titanic acid will thus be completely precipitated.

Or - Through the filtrate from SiO_2 pass H_2S until smells strongly; filter and boil for several hours to separate TiO_2 - Filter dry, ignite and weigh as TiO_2.

Granite - Mineralogical Analysis.

Quartz	—	40.97 o/o. -
Felspar	—	52.57 "
Mica	—	6.46 "
		100.00 "

From this the following analysis is estimated by taking averages of a large number of analyses of Quartz, Felspar & Mica: -

Analysis of Granite: -

$Si O_2$	—	78.55 o/o
Al_2O_3	—	11.75 "
Fe_2O_3	—	.23 "
$Mg O$	—	.15 "
CaO	—	.33 "
Alkalies	—	8.71 "
Combined water	—	.26 "

Specific Gravity = 2.671 —

Resists action of conc. HNO_3 & Sol. well - also but little of it is dissolved in cold HCl. Heated and dropped into cold water, it breaks into many pieces.

Felspar. – Average analysis of. –

SiO_2	——	65.61 o/o.
Al_2O_3	——	18.37 "
Fe_2O_3	——	.17 "
MgO	——	.14 "
CaO	——	.57 "
Na_2O	——	1.39 "
K_2O	——	13.75 "
		100.00

Mica Average analysis of. –

SiO_2	——	45.89 o/o
Al_2O_3	——	33.44 "
Fe_2O_3	——	3.70 "
MgO	——	1.18 "
CaO	——	.46 "
Na_2O	——	1.90 "
K_2O	——	9.42 "
H_2O	——	4.01 "
		100.00

Quartz.

SiO_2 = —— 100.0 o/o. –

These average analyses were obtained from Dana.

Granite.

To make mineralogical analysis, coarsely powder and as nearly as possible separate mechanically into its three constituents - Quartz Felspar & Mica.

Water gas - (Illuminating) -
Analysis.

	Vols. per 100.
Carbonic acid (CO_2)	0.11
Heavy Hydro-carbons	10.30
Oxygen	.59
Carbonic Oxide (C_2O_2)	28.36
Hydrogen	44.73
Marshgas	14.30
Nitrogen	1.61
Sulphuretted Hydrogen	0.00
	100.00

Density — 0.630 r

Course of analysis. -

Apparatus used - Hempel's gas - burette, absorption pipette, and Explosion pipette.

The gas-burette were filled with gas, great care being taken to have all connecting capillary tubes filled with water. The burette was filled to the mark 100.

CO_2. A solution of KOH was introduced into the burette to absorb CO_2. After absorbtion was complete, the solution in the two tubes were brought to the same level and the amount of gas left read off. - Difference = CO_2.

It would really be preferable to absorb this CO_2 in the absorption pipette filled with KOH. - The gas can be easily transferred from the Burette to the Pipette and back. If gas contains H_2S, this should be absorbed with slightly acid solution of $CuSO_4$ (use H_2SO_4). -

Heavy Hydrocarbons (C_nH_{2n}) and (C_nH_{2n+2}).

Gas remaining after absorbing CO_2 is transferred to absorbtion Pipette containing Nordhausen Sulphuric acid (Fuming). This absorbs the heavy hydrocarbons. When absorbtion is complete gas is again

transferred to Burette for measurement. difference = Heavy Hydro-Carbons.

Oxygen. - Determined by absorbing in alkaline (Potash) solution of Pyrogallic acid, which can be accomplished in Burette direct. -

Difference = O. -

Carbonic Oxide (CO or C_2O_2). Determined by absorbtion in Burette with ammoniacal solution of Sub-Chloride of Copper. Before reading, gas should be washed with dilute H_2SO_4 to absorb any ammonia left in it from Copper solution.

Difference = CO.

Marsh Gas and Hydrogen; by Explosion. See Hempels "Analysis of Gases" and follow closely. — The proportions of Hydrogen and Marsh gas were obtained from following proportions: -

$$x = O - \frac{C}{3} \quad \text{and} -$$

$$y = \frac{4C}{3} - 2O.$$

x = Marsh gas.

H = Hydrogen.
C_1 = Total contraction.
O = Oxygen used in Explosion.

Nitrogen. - By difference - i.e. what is left of 100 after all other gases have been determined.

Density. Determined by inserting a glass tube with a very small aperture in the upper end of gas burette, and comparing the time required for equal volumes of of gas and air to pass through this small aperture under equal pressure. The specific gravities of two gases are to each other as the square of the time required for each to pass through same aperture.

References -
Analysis - Hempel and Winkler.
Density - Hager - Vol. II. p. 41. -

Hydrogen. Determination of in gas (after removing H_2S, CO_2 C_xO_x, Heavy Hydrocarbons) by absorption with Palladium asbest.

This is, up to date (1889) by far the best method known. (Le Winkler.)

Apparatus – Hempel's gas-burette + gas pipette, capillary tubing and Palladium-asbest. –

Into a capillary tube about 6 inches long is introduced a Palladium thread about an 1½ inch long. This can easily be accomplished by placing 3 or 4 threads of asbest side by side and to the length of 1½ inch on a piece of filter paper, moistening with a drop or two of water, and, by twirling and rubbing backwards and forward, is made into a fine thread. This is introduced into the aperture of the tube, and by shaking or with the aid of water, is brought to the middle of the tube. The tube is then dried in a warm place. Then

at about ~~att~~ 1/2 inch from each end bend the tube at right angles.

Leave about 20 & 24 c.c. of gas in the burette and fill up to 100 with air - noting how much air is added. Fill the pipette full of water. Then connect one end of tube containing Palladium each with burette and pipette. Now for 3 to 4 minutes heat, with a small Bunsen-burner flame, that portion of the tube which contains the Palladium. - Then by raising the "Niveau" tube, the gas is forced ~~by~~ in a slow current through the capillary tube over the Palladium. The tube is kept heated with the burner. - The heat should never be raised to redness or any where near it; but just to a faint, dull tinge of red. The reaction of oxidizing the H is so energetic that the end of the Palladium toward the current of gas, glows like a spark. The gas is then, by lowering the "Ni-

vacuum tube, transferred back to the burette in the same way. —

This operation is gone through a second and a third time, if necessary until there is no longer a diminution in the volume of gas.

2 volumes of H combine with 1 vol. of Oxygen to form H_2O. —

Hence 2/3 of the decrease in volume = Hydrogen.

If gas is a mixture of Hydrogen and Marsh gas use in pipette and burette, KHO solution instead of water; and determine Oxygen left in gas after absorption. Marsh gas is oxydized much more slowly than H. and perhaps not completely. (Look up this point).

Then determine proportions of H + Marsh gas from following proportion

$$x = O - \frac{C}{3}.$$

$$y = \frac{4C}{3} - 2O.$$

X = Marsh gas.—
Y = Hydrogen.
C = Total Contraction.
O = O consumed.

Air contains 20.75 & 21. % O.

Sp. Gr. and absolute weight of some gases.

	Sp. Gr.	wt. of 1 litre at 0°C. and 760. m.m. pressure. grammes.
Atmospheric air	1.00000	1.29366
Oxygen	1.10832	1.43379
Hydrogen	0.06927	0.08961
Water, vapor of -	0.67343	0.80651
Carbon Dioxide —	1.52394	1.97146
Carbon Monoxide	0.96978	1.25456
Marsh Gas	0.55416	0.71689
Elayl gas	0.96978	1.25456
Hydrosulphuric acid (H2S)	1.17759	1.52340
Iodine, vapor of	8.78898	11.36995
Bromine, vapor of	5.53952	7.16625
Chlorine	2.45631	3.17763
Nitrogen	0.96978	1.25456
Ammonia	0.58879	0.76169
Cyanogen	1.80102	2.32991

Milk. Analysis.

Total Solids	10.93	%.-
Water	89.07	"
Fat	1.83	"
Caseïn	4.07	"
Albumen	.30	"
Cream	4.68	% by volume.

Total Solids. - See p. 79.

Water - by difference.

Fat. Extract total residue with Ether. Ether extract is brought to dryness in small tared beaker on water bath and weighed. - Residue = Fat.

Caseïn. - Take 20 cc. of the well shaken milk and dilute to 200 cc. - Then add drop by drop, very dilute acetic acid, until a flaky, white ppte. begins to form. a current of CO_2 gas is then passed through solution for about 25 minutes, and allowed to stand for 24 hours. The Caseïn with Butter is thus precipitated. Filter and extract

butter fat with alcohol and then Ether. The Casein which now remains on the filter (Fund) is dried at 110° C and weighed.-

Albumen.- The filtrate from the Casein is heated to boiling the Albumen collected on a Fund filter, dried at 110° C. and weighed.

Cream. Fill test tube to the height of eight inches with the well shaken milk and allow to stand for 24 hours. At the end of that time the height of the column of cream is measured.

Formula for Calculation of Solids
Let S = Sp.G.
f = % of fat
x = Solids not fat - then

$$\left[\frac{S.100 - S.f.}{100 - 1.00753\,S.f.} - 1\right] \times (100 - f)2.6 = x.$$

Lead - analysis.

Pb	99.1300 o/o. -
Fe	.1070 "
Zn	.4200 "
Sb	.2850 "
Ag	.0008 "
Total	99.9428 -

Current analysis. -

1 to 4 grams lead taken, dissolved in dil. HNO_3 brought to dryness and dissolved in H_2O. Collect the residual Antimoniate of Lead on a filter, saving filtrate (a).

Dissolve residue in HCl, neutralize with NH_4OH add $(NH_4)_2S$ to precipitate Sulphide of Lead, Sb remaining in solution; filter. Filtrate (b) -

Lead sulphate ppte. should be dissolved and added to filtrate "a".

Sb. In b ppte. Sb as sulphide by adding excess of dil. H_2SO_4. filter. The Sulphide is then converted into the

Oxide of Antimony by treating with fuming Nitric acid, and bring to dryness in a platinum dish (tared). Ignite to a dull red heat until the weight remains constant, and weigh as Sb_2O_4.

Pb. Determined in filtrate "a" as given on p. 37.

Fe. Expel alcohol largely from filtrate from $PbSO_4$, nearly neutralize with NH_4HO, add sodium acetate and boil to ppte. Fe. Filter. Ignite residue + weigh as Fe_2O_3.

Zn. In last filtrate, ppte. Zn as sulphide with H_2S, filter and wash. Dissolve ZnS in smallest possible amount of HCl and ppte. with Na_2CO_3 or K_2CO_3. Wash very thoroughly, dry, ignite in porcelain crucible and weigh as ZnO.

Ag. Take 50 to 100 grams lead and determine by cupellation and cupellation.

Nitrogen. Determination in fertilizers and other organic matter.

Kjeldahl's "New Method" - (Neue Methode)

1/10 gram or more of ~~finely~~ powdered sample placed in glass flask of about 100 cc. capacity. Add 10 c.c. conc. H_2SO_4 and heat on wire gauze over a flame. When strong effervescence has subsided, add a small amount of Phosphoric anhydride, and keep solution at about the boiling point until it is nearly colorless, of a slight yellowish tinge. Oxidation is then effected by adding finely pulverized Permanganate of Potass. to the hot solution, it must be added in very small quantities at a time, as the action is very energetic, and in excess to insure complete oxidation. The color of the solution will serve as indicator in this matter; when the solution has assumed a bluish green color, oxidation is complete and sufficient Permanganate of Potass. has been added.

Leave flask over the flame a few minutes longer and then allow to cool. Dilute with water; on doing this the green disappears and the solution turns brown.

Now introduce it into a flask of about 3/4 litre capacity, close flask with perforated rubber stopper and connect with Liebig's condenser, the exit tube of which is introduced into a bottle containing 20 cc. of Normal Hcl or H_2SO_4; - it was not however allowed to dip into the ~~bottle~~ acid.

Strong solution of KHO or NaHO, after thoroughly boiling it to expel all possible N_2 is, after introducing a few chips of Zinc into the flask, added in excess to the H_2SO_4 solution in the flask, and the flask quickly closed with stopper to prevent loss of NH_3. Boil, distilling NH_3 over into Normal acid solution. Boil until NH_3

has all passed over or until solution in flask bumps so that distillation must be discontinued. –

The amount of acid neutralized by NH_3 so distilled can be determined by titrating with Normal NH_3 solution; and so the N can be estimated. Or the NH_3 in HCl sol. can be determined by adding Platinic chloride Ppte. NH_3 – Evaporate almost to dryness and add alcohol. – filter and wash with dilute alcohol (80%) on tared filter. Dry and weigh as $(NH_4)_2PtCl_6$.

Soft Coal. Analysis. —
Samples from Illinois. —
"St. Davis."

Water — —	11,37 o/o
Volatile Matter	35,81 "
Fixed Carbon —	50,41 "
Ash ——	2,39 "
Sulphur ——	1,18 "
Heating Power —	7356.6 Calories.

"Cleveland Hill."

Water ——	12,85 o/o.
Volatile Matter —	31,40 "
Fixed Carbon —	53,57.
Ash ——	2,16 "
Sulphur	1,706 "
Heating Power —	7757. Calories.

Rapids City, Ill.

Water	11.72 %.
Volatile Matter	28.01 "
Fixed Carbon	51.75 "
ash	8.97 "
Sulphur	2.30 "
Heating Power	6840. Calories.

Course of analysis.

Place 3. to 5. grams of the finely pulverized coal into Porcelain or platinum crucible and dry at 110° to 115° C. - Weigh - Diff. = Water, Volatile Matter. The crucible containing the dried coal is covered and ignited strongly until all volatile matter has passed off. Coal and weight. Diff. = Volatile matter.

Fixed Carbon & ash. Remove lid and ignite. - Diff. = Fixed Carbon. Residue = ash.

Sulphur. Take one (1) gram finely powdered coal and mix with 8 grams

If a mixture of equal parts of Na_2CO_3 & KNO_3 fuse coal, dissolve in water & HCl, filter, solution contains S as Sulphate. Precipitate with $BaCl_2$ in usual manner and estimate as S.

Heating Power - To estimate theoretically. -

Heating power of 1 pt. carbon = 8000 calories.

" " " 1 " Hydrogen = 34,000. "

The average amount of H in soft coal is 5 %, in use this figure in estimation of all samples. The average amount of Carbon in volatile matter is equal to the percent of volatile matter less 15.5 -

The amount of carbon thus obtained added to the Fixed Carbon = Total Carbon.

Let x = % of Total Carbon

" y = " " Hydrogen -

Then -

Heating Power = 8000 x + 34,000 y.

As an example take "Cleland, Ala," of which an analysis is given above.

31.40 = % Volatile Matter.
15.50
15.90 = % C (in Volatile Matter).
53.57 = % Fixed Carbon.
69.47 = % Total Carbon.
5. = % Hydrogen.

8000. × .6947 = 5557.6 Calories from Carbon.
34000. × .05 = 1700. " " Hydrogen.
Heating power = 7257.6 Calories (Total).

Heating Power should be determined by actual test.

Calories × 9/5 = B. T. U. or × 1.80

Method of Test for B. T. U.

1 Gm of sample mixed on glazed paper with 30 gms KClO3. Transfer to Berlin crucible and cover with 20 gms KClO3 + a crucible cover. Heat for 2-3

Method B. T. U.

minutes. Remove coal and weigh. Duplicate assays should not vary over 0.025 grms.

Calculate results as follows:

1 Pt Ca produces 34 grms from Pb O.

8080 = Calories of C. = a.

b = wt Pb produced in assay

C = grms Pb produced by given Calories

then:

$$\frac{a}{C} \times b = \text{X Calories per 1000 grms}$$

$$X \times \frac{9}{5} = \text{B.T.U.}$$

Example:

$a = 8080$
$b = 31.5$ $\Big\}$ $\therefore$ $\frac{8080}{34} \times 31.5 = 7485.8$
$C = 34.$

$\frac{7485.8}{5} \times 9 = 17674.4$ B.T.U.

Correction

$\frac{a}{3}$ = factor of Calories 322 = factor for N.

Nickel. Determination of - in ore. -

Take 1. to 3. grams of finely powdered ore and carefully roast, adding charcoal and Carb. of Ammon. Toward the end of the operation. Boil the roasted ore with aqua Regia and bring to dryness on water bath. Add HCl and water; pass H_2S through solution and filter. Boil filtrate with a few crystals of $KClO_3$ until H_2S is all expelled, and bring to dryness on water bath until HCl has been completely driven off. Take up residue with water and filter. To filtrate add excess of a neutral solution of Oxalate of Potass. Then add strong acetic acid about equal in bulk to the solution containing the Ni.

The Nickel (+ Cobalt) will thus be precipitated. Heat to 50° or 60° C. Filter and dry. Remove ppte. to a Platinum Crucible, burn filter

and add ash & crucible, and cover the latter. Heat – at first slowly, gradually raising the temperature. Finally remove the cover and ignite strongly. Weigh as Ni_2O_3.

In event of Ca being in the ore this must be removed before the Ni is ppt'd.

Soap. Determination in — of Rosin and Fatty acids.

Analysis of sample —

Fatty acids ——	60.76 %
Rosin ——	8.46 "
Sum ——	69.22 "

Process. — The two are first determined together. Take 10 grams soap, dissolve in 50 c.c. dist. water. Acidify solution by adding drop by drop about 6 c.c. HCl (Sp. gr. 1.124). Acids rise to the top — allow to cool, and they harden. Filter and wash on a Hard filter. Transfer filter paper and contents to a Hard beaker and dry at 100° C. until a constant weight is obtained. Weigh Fatty and Rosin acids. To get amount of anhydrous acids from this weight, deduct 3¼ % of weight.

Rosin. — The mixture of these acids (Fatty about 1 gram) or an aliquot part, is dissolved in 20 c.c. alcohol and saponified with NaHO in slight excess. Boil, allow to

cool and fill up to 100 cc. with Ether, add finely pulverized $AgNO_3$ in excess and shake for 10 minutes. allow to settle. The solution contains compound of Ag and Rosin acid. Of this solution 25 cc. — Wrn Fasten, 20 cc. of HCl (1:50) added, the AgCl allowed to settle and an aliquot portion of the Ether solution evaporated in a Tared beaker.

Residue dried at 110° C. and weighed as Rosin acid.

Deduct Rosin acid from total acids to get Fatty acids.

Another method is based on fact that Fatty acids are soluble in alcohol while Rosin acids are insoluble — but as latter are not entirely insoluble, method is not very satisfactory.

Pig Iron. Determination of P. S. & SiO_2

P ——— 0.1404 o/o.
S. ——— 0.0193 "
SiO_2 ——— 0.7842 "

P. - Dissolve 2 to 4. grams sample in Aqua Regia, then bring to dryness on water bath in Porcelain dish. When dry - add HNO_3 and dry again - repeat this opperation until HCl is expelled. Dissolve residue in HNO_3 + H_2O - filter. Retain residue (a) for SiO_2. - In filtrate determine P with Ammon Molybdate, as described in analysis of Iron or Steel.

SiO_2. - "a" fused with Na_2CO_3 & KNO_3 - dissolve in H_2O + acid - brought to dryness - and SiO_2 separated in usual manner. -

Sulphur. Place 10. grams Pig Iron in a flask. Supply flask with a doubly perforated cork through which pass an exit tube and a Brit funnel

or thistle tube. The exit tube is connected with two U tubes (or with bulbs) but U tubes are preferable. These contain a strongly alkaline solution of lead, prepared by adding NaHO or KHO to solution Acetate of lead. The thistle tube contains a little Mercury in the bend. HCl is now poured into the thistle tube and drawn into the flask by applying suction at the end of the 2nd U tube. Apply a little heat and add HCl from time to time until effervescence has ceased and solution is complete. Attach an aspirator to U tube, and draw air through apparatus for some time. Filter lead solution in U tube dry the residue ($PbSO_4$) and carefully fuse with Na_2CO_3 + KNO_3 in porcelain crucible. — Dissolve in H_2O + HCl — filter and ppte. SO_3 with $BaCl_2$.

Wall paper. Test for arsenic.

Introduce a sample of the paper directly into the Marsh apparatus. If paper effervesces with acid – it ought to be moistened with H_2SO_4 before it is put into apparatus.

Axle Grease — Frazer's.

Composed of —

1/2 lb. Sal Soda.
3. pts. Water.
17. Gals. Rosin Oil.
10 lbs. Sifted Slacked Lime

Fire Solutions for hand grenades.

No. 1.

Sesqui carbonate of Ammon.	2. lbs.
Water	1. gal.

No. 2. Bi carbonate of Soda. 1½ lbs.
Water 1. gal.

No. 3.

Bi-Sulphite of Soda	2. lbs.
Water	1. gal.

No. 4.

Bisulphite Soda	2. lbs.
Weakest common Aqua Am.	1. gal.

No. 5.

Bi-Sulphite Ammon.
Saturated Solution of.

Other Solutions.

1. Solution of $CaCl_2$
2. " " $CaCl_2$, $CaSO_4$ + NaCl.
3. " " $CaCl_2$ NH_4Cl, NaCl + NH_3

Hair — Solution for dying —

$AgNO_3$ —— 1.49 %

$CaSO_4$ —— .27 "

Water q.s. —— 100.00 ——

NH_4HO —— small amt.

Solution of gallic acid applied to hair before using above.

Whiskey. Examination for artificial coloring matter. —

Test for Sugar (caramel or burnt sugar) after ~~adding~~ coloring matter with Acetate of Lead.

Test for ~~Tannic~~ Tannic acid with Ferric chloride, glue, and Albumen.

Determined Sp. gr. (990.)

Also Solid Residue — = 0.107 % which is very low.

"Beef Peptonoids" or "Prepared Food."
Analysis —

Bone Phosphate	2.49 o/o
Other Mineral salts	4.24 "
Fat	7.67 "
Dextrin	5.78 "
Starch	3.71 "
Moisture	10.02 "
Albuminoids	65.23 "
Cellulose	.69 "

Course of Analysis.

Total Min. Salts. Ignite a screwon portion and weigh ash. —

Bone Phosphate. Boil 1 gram with HCl + HNO3 — add excess of NH4HO to filtrate and ppte. P2O5 with Mag — nesia mixture. Weigh as Mg2P2O7 — Estimate Bone Phosphate + deduct from total salts for "Other Min. Salts."

Fat. Extract with Ether, Evaporate and weigh extract (Fat).

Dextrin (or Milk Sugar). Extract

from "food" with cold water, boil extract with HCl + determine Glucose with Fehling solution. see p. 3. etc.

Starch. The residue after extracting with cold water, boiled with HCl or H_2SO_4, add excess NaHO, and determine glucose. (Fehling)

Celulose. - Residue after boiling with HCl is collected on hard filter, dried and weighed as Celulose.

Moisture. By drying known amount at 110° C.

Peptones (albuminoids) Ignite 1 gram with Soda lime in combustion furnace (tube) and collect NH_3 in Nor. HCl. Determine NH_3 either by titrating with Nor. NH_4HO or by precipitating ~~with~~ as $(NH_4)_2PtCl_6$. From N (NH_3) estimate amount of Peptones.

Axle grease. Frazer's.

Analyses.

1. Superior axle grease.

Mineral Res. (Lime)	6.87 %
Water	4.91 "
Palm Oil	.70 "
Rosin Oil	87.52 "
Sum	100.00

Sal Soda 10 Lime 15 de Frazer

2. Frazer Axle Grease.

Mineral Res. (Lime)	5.46 %
Water	2.06 "
Palm Oil	1.73 "
Rosin Oil	90.75 "
Total	100.00

Sal Soda 10 Lime 15 de Frazer.

Course of analysis.

Palm Oil. - Place weighed sample in flask and pour 20 times its bulk of 85 to 95 % alcohol over it. Shake up and macerate for a day or two. Pour off alcoholic extraction and wash with alcohol. - The residue will

consist of mineral matter (lime) and Palm Oil, while alcohol dissolves Rosin oil. Now extract Palm oil from residue with Ether, and evaporate Ether extract in a tared vessel. Weight = Palm oil.

Mineral Residue. Heat sample to thin fusion — Lime will settle to bottom, pour of oil through hot filter — wash with alcohol & Ether, and ignite and weigh residue. — Or — Lime can be extracted from grease by heating and shaking with dilute acetic acid and a few drops of HNO_3. Draw off acid solution (or filter) add NH_4HO in excess, also NH_4Cl & ppte with Oxalate of Ammon.

Water. — Cannot well be estimated by heating as volatile oils go off. On some oils it will do, but not in all. It can be done by placing grease for some time over H_2SO_4

and $CaCl_2$ in dessicator for long time and thus drying.

Rosin Oil - Estimated by difference. Soda (Sal) was too small to be determined accurately.

Linseed Oil. Examination for adulterations.

See Tables for various color tests etc. etc. Thayer. —

Determine Sp. gr. —

Also freezing point. —

Cottonseed oil solidifies at 1. to 3°. C.

Linseed " " " -15 to -25°C.

Rosin (Colophony). — Extract oil with alcohol and test for Rosin by adding alcoholic sol. of Acetate of Lead to extract. If Rosin present a cloudy white ppt. will form.

Tests for Mineral Oil — the most common adulterant. —

1. — Taste and odor.

2. On heating suspected oil, if it contains mineral oil, its flash point will be lower than that of pure linseed oil. Flash point of pure oil is 300° C. To test this, place oil in platinum dish and heat direct with flame. From time to time hold burning

match over surface of oil, when the point is reached where fumes are given off which explode or ignite, take temperature.

3. Saponification. Mineral oil does not saponify, and a quantitative determination can be made in this way, as follows:

Place 10 grams of oil in flask with a narrow neck; add 50 c.c. of strong solution of NaHO and heat for several hours on water bath, shaking from time to time. When saponification is complete, fill flask with water and heat until soap is dissolved. The mineral oil will rise to the top and into the narrow neck and can be drawn off with a pipette and with the aid of a little ether can be completely transferred to a tared beaker & weighed.

4. - Rise in temperature when

mixed with H_2SO_4. Test as follows:
(Maumé's test) Place 50 grams of oil into rather a spacious beaker, surround this with cotton and place it into a larger beaker, so as to prevent loss of heat. Take temperature of oil — then add to it 10. c.c. conc. H_2SO_4 and stir with thermometer. Note the highest point reached by mercury.
Pure linseed oil should rise 133° C in temperature, higher than any other oil.

If no other oil is mixed with linseed oil to affect rise in temperature an approximate quantitative determination of mineral oil can be made. For example a suspected oil rises 100° C. or 33° less than pure oil. — Then from this proportion

133 : 33 :: 100 : 4 = Mineral oil.

Rosin if present in considerable quantity can also be detec-

quantitating by saponifying with K_2CO_3 (or Na_2CO_3) and separating with NaCl + HCl.

All color and other similar tests should be compared with the same tests with an oil of known purity.

Analyses of two oils —

1. —

Mineral oil —	20.27 %
Rosin —	1.00 —
Linseed oil —	78.73

2.

Mineral oil —	22.55
Linseed oil —	77.45

Pure Linseed	Iod value	mean	170.
	Sap. no	"	197
	Maumn	"	133
Cotton oil	Iod value	"	101
	Sap. no	"	192.5
	Maumn		74–75

Zinc, determination in Carbonate and Sulphide ore.

Take one gram finely powdered, place in porcelain dish, add 10 cc. HNO_3, 5 cc. HCl, & 10 cc. H_2SO_4 (all conc.). Bring nearly to dryness; add HCl & water, & saturate with H_2S, and filter. Boil solution with a few crystals of $KClO_3$; very nearly neutralize with Na_2CO_3 or K_2CO_3, add Sodic acetate in excess; boil & filter to separate Fe & Al. & add acetic acid to filtrate. ppte. Zn with H_2S, allow to settle, filter, dissolve in HCl & ppte. Zn. with Na_2 or K_2CO_3. Filter, wash very thoroughly and ignite. Weigh as ZnO.

1. Dry Bone ($ZnCO_3$) contained 39.87 % Zn.
2. Blackjack (ZnS) " 60.62 " "

Glycerine - Examination as to purity. Not a pure sample.

1.- Sp. gr. = 1.2203 -

Represents -85,72 % Glycerine.

Glucose — none.

Butyric acid - trace.

Gums — trace.

Albuminoids - none.

Phosphoric acid - present.-

HCl, H_2SO_4 — "

Ca, NH_3 — "

Mineral Residue - .4915 %.

Ca — .021 "

Tests - Glucose with Fehling's Sol.-

Butyric acid - H_2SO_4 + alcohol.

Gums - extracting with 4 vol. mix-

ture Equal pts. alcohol + Ether.

Albumen.- Mineral acids alkalies,

Dextrine, which ppte it.

Acids etc. ordinary tests.

Mineral Residue. Ignite 20. grams.-

CaO — . In Residue. -

In most test use water solution.

Glycerine. Examination as to purity. (a Dunn sample.)

No Sugar. Tested with KHO + Fehling's sol.
No HCl " " $AgNO_3$. (H_2O sol.)
No H_2SO_4 " H_2O sol. with $BaCl_2$.
No Butyric acid. – Tested with H_2SO_4 + C_2H_6O.
No NH_3 – Tested with KHO + heat.
No Gums, " by extracting with mixture of equal pts. alcohol + Ether.
Free Oxalic – tested H_2O sol. with $(NH_4)_2C_2O_4$.
Reaction neutral.
~~Ether~~ Dissolves clear in 2 pts. alcohol. and one(1) pt. ~~Ether~~.
Sp. gr. = 1.251

Baking Powder. (Prices)

Analysis.

Available CO_2 —— 11.91 % (11.58)

Unavailable CO_2 —— none. "

Starch —— 17.16 "

NH_3 —— none.

Reaction slightly acid. -

11.58 % CO_2 = 107.1 cc. inches available CO_2 per ounce.

Course of analysis.

Available CO_2. Introduce weighed quantity of powder into a dry CO_2 apparatus; and water into upper portion; weigh apparatus - bring powder and water together; available CO_2 will pass off. Heat apparatus & expel CO_2; cool and weigh. Diff. = available CO_2.

Unavailable CO_2, introduce Helen H. Say into apparatus, to expel any CO_2 left combined. Difference, if any = unavailable CO_2 —

Starch. Treat and gently heat, (not high enough to gelatinize Starch) a weighed sample of powder with water. Filter through Faraday filter, wash, dry at 100° to 110° C and weigh starch.

H_3 – Test with Nd HO.

Do test for reaction of powder boil filtrate from starch and test with litmus paper.

Butter Color. - Recipe for - obtained after many experiments.

1. Add 1½ lbs. Annato seed to 1 gal. of a mixture of 1 part of Castor oil and 3 pts. of Linseed oil (must be absolutely pure, not boiled). Heat for 3 or 4 hours to a temperature of 175° F, which gradually increase to 200° & 210° F. toward the end.

2. Separately add 1½ lbs Curcuma to one (1) gal. of a mixture of 1 pt. Castor oil and 3 pts. Linseed oil. Heat same as No 1.

Now mix 3 parts of No. 1. (Annato color) with 2 parts of No. 2. (Curcuma color). This will give the desired color.

No 1. & 2 should be strained either before or after mixing.

The strength is not perceptibly diminished by mixing the two colors.

No. 1. & 2 should each be saturated with coloring matter before mixing. The Annato and Curcuma of Commerce not being uniform products, varying in strength, larger or smaller amounts may have to be taken from different batches to saturate the oils.

By changing the proportions of No. 1. & No. 2. the shade of color may be greatly varied.

Paint-White- Analysis of mineral constituents.

$2PbCO_3 + Pb(OH)_2O_2$ (White Lead)	18.3 % o/o
$CaCO_3$ ———	2.01 "
$BaSO_4$ + Clay ———	33.88 "
$PbSO_4$ ———	1.28 "
ZnO ———	43.97 "

Course of analysis.

a Sample of the "settling" (solid matter of the paint) is placed in a flask and thoroughly digest with Benzine (or ether). filter, wash with Benzine (or ether) and dry.- Of this the analysis is made.

Take 1. grams of this residue and heat in a covered dish with dilute HNO_3 and concentrate to a small volume. Add alcohol to throw down $PbSO_4$ in paint. Filter and wash with mixture of alcohol + water. (Filtrate "a").

$PbSO_4$. Stop the funnel containing last residue with a cork and pour $(NH_4)_2S$ on the filter. Cover funnel and allow to stand for ½ to 1 hour. This converts all $PbSO_4$ into PbS. Remove cork and wash thoroughly with H_2S water. Throw away filtrate. Now pour hot dilute HNO_3 on filter & dissolve PbS; wash, and $BaSO_4$ + Al_2O_3 are left on filter. Ppte. Pb in HNO_3 solution in usual way as $PbSO_4$.

$BaSO_4$ + Clay. These can be estimated together by igniting residue left after washing with HNO_3. - Or they can be separated by fusing with Na_2CO_3 + small amt. of KNO_3, dissolving in water, filter. In filtrate determine SO_3 by ppting. with $BaCl_2$. Dissolve residue in HCl ppte. Ba with H_2SO_4 + then Al_2O_3 with NH_4HO.

White Lead ($2PbCO_3 + Pb(H_2O_2)$).
Precipitate Pb in Filtrate "a" with H_2SO_4 in usual way. Filter and wash. (Filtrate "b.") Weigh as $PbSO_4$ and estimate as $2PbCO_3 + Pb(H_2O_2)$.

Zn O. After expelling the alcohol largely from filtrate "b," add Sodium acetate and ppte. Zn as Zn S with H_2S. – allow to settle, filter and wash. (Filtrate "c").
Dissolve Zn S in small amt. HCl and ppte. with Na_2CO_3 or K_2CO_3. Filter wash thoroughly dry, separate ppte. from filter, incinerate filter in Porcelain Crucible, add ppte. and ignite. Weigh as ZnO and estimate as such. –

Whiting ($CaCO_3$). Concentrate filtrate "c," add excess of NH_4HO and ppte. CaO with Oxalate of Ammon. –

O_2. Determined with separate sample in O_2 apparatus.
The White Lead ($2PbCO_3 + Pb(H_2O_2)$) can

also be estimated from amount of CO_2, after deducting CO_2 in $CaCO_3$.

Cider - analysis. Determination of Total solids, alcohol and water in.

Analyses of 2 old samples. -

1. - Total solids —— 2.23 o/o
Alcohol —— 2.40 "
Water —— 95.37 "

2. -
Total solids —— 1.57 o/o
Alcohol —— 3.33 "
Water —— 95.10 "

Analyses of two new samples:

3. -
Total solids —— 3.70 o/o
Alcohol —— 1.30 "
Water —— 95.00

4.
Total solids —— 7.25 o/o
Alcohol —— 2.55 "
Water —— 90.20 "

Course of analysis. - Total solids are

determined by evaporating a known weight to dryness on water bath in a tared Platinum dish, drying at 100° to 110° C. and weighing. –

Alcohol. Place 100. c.c. in a retort, add NaCO3 to neutralize acetic acid and distill. Measure distillate, take Sp. gr. and estimate alcohol from Table of alcoholimetry.

Water. By difference. This is not really correct as it will include acetic acid and other volatile matters, but it is near enough for ordinary purposes.

"Tolu Rock & Rye" analysis. —

1. { Alcohol — 24,15 o/o
   { Balsam Tolu 1.56 "

2. { Alcohol — 28.78 o/o
   { Balsam Tolu .86 "

3. { Alcohol 24.59 o/o
   { Balsam Tolu 1.83 "

4. { Alcohol 27.09 o/o
   { Balsam Tolu 1.09 "

Source of analysis.

Alcohol — determined by distillation.

Tolu Balsam determined by Evaporating December to dryness. Extract with water (to dissolve sugar) filter, wash, dry & weigh. This weight less residue in whiskey used (approximate) = Balsam Tolu.

Linseed oil solidifies at ~~+1° to~~ -15° to -27° C.

Cotton seed oil solidifies at +1° to +3° C. -

Difference in rise of temperature when mixed with H_2SO_4.

Burnishing Size.

Analysis. —

Water	——	50,760 %
Clay	——	43.408 "
Graphite	——	4.920 "
Fat	——	.504 "

Source of analysis.

Water by drying at 100° to 110° C.

Fat - by extracting with Ether.

Graphite by direct ignition.

Clay = weight after burning.

Assaying. —

Gold & Silver.

Ore consisting of Quartz, or basic ore containing little or no Sulphur, Antimony or Arsenic (or Copper) I prefer to assay by crucible process. —

Ore containing considerable quantities of S, Sb, As or Cu I prefer to scorify.

If it is desired to make a crucible assay of ore rich in Sb, or As then samples should be roasted before mixing with fluxes. An ore containing S can be treated in crucible without roasting by sticking 2 or 3 iron nails into the charge.

Galena ore should always be scorified in preference to crucible assay. —

Crucible Operation. - Use Battersea

or Colorado B cruba, they be-
ing a convenient size.

1. Charge - ore consisting largely
of Quartz or other Silicacious rock.

Ore	1/2 A.T.	1. A.T.
Litharge	1 " "	1 1/2 " "
Bi carb. Soda	1 1/2 " "	2 1/2 " 3. " "
Powd. Charcoal.	1/2 " 1. Gram.	1/2 " 1. gram.

V. Basic ore (containing $CaCO_3$, Al_2O_3
Fe_2O_3 etc. in considerable quantity.
Same proportions as 1. about the
same, but add about the same
quantity of Quartz sand as of ore. In-
crease amt. of Charcoal.
3. - In an ore containing Sel-
lurium use double the am-
ount of litharge given in 1.

4. In ore containing Sulphur
add three times as much as 1. -

5. In ore containing large

amount of Iron dust & powdered charcoal.

6.- Galena - fuse with Black Flux Substitute (mixture of flour and bicarb. of Soda) and Bi-carb. Soda.- Add a little Litharge if necessary that is if Lead in ore is to small to give large enough button.

Each charge is covered with 1/4 inch of salt (Nacl) -

Place crucible in furnace and heat until mass is in a quiet state of fusion. Then remove and allow to cool. When cold break crucible and remove button of lead. Cleanse button by hammering. Button should not be quite as heavy as the copel below used. If heavier, scorify down to proper size.

Scorification.

Charge —

Ore —— 1/10 to 1/5 a.t.

Granulated Lead — 2. a.t.

Borax Glass. ——

Place 1. a.t. of Lead in scorifier and add ore; mix thoroughly, then spread 1. a.t. Lead over the top. Place a few little pieces of Borax Glass on surface of charge.—

If the ore is free from S, Sb, & As & Zn, place into a hot muffle. If ore contains S, Sb, As &c. place in moderately hot muffle and gradually increase heat; thus roasting ore before scorification proper begins. and closed

Keep muffle at high heat until fusion is complete. Then open muffle, reduce heat a little, and continue oxidizing

until slag formed completely covers the charge - Remove and pour into a mould. Cool and Separate Lead button. If button is still too large scorify again.

Cupellation. - Place a bone ash cupel into muffle and heat to dull if it does not crack. Then put lead button (from crucible or scorifier) into it, close muffle and heat until completely fused. Then open muffle and oxidize until all the lead is oxidized and absorbed by cupel, and only the bead of Au+Ag is left. Heat for a few minutes longer and remove cupel. Cool+weigh bead. If it contains Cu, treat bead with dilute HNO_3 to dissolve Ag, wash Au, dry, ignite and weigh. If bead contains more than 20 or 25. % of gold, it should be

fused with pure granulated silver, before a blowpipe on a piece of charcoal, before parting with HNO_3. —

1. mmg. of Au or Ag in 1 A.T. of Ore represents 1. ounce of Au or Ag in 1. Ton of ore.

Cupels — to make —

Take equal parts of the finest powd. boneash, and of bone ash ore grade coarser, and mix. Add a weak solution of K_2CO_3 and mix. Enough of the solution should be added so that a portion of mixture, when pressed together in hand, retains it form. Care must be taken not to get it too wet. — Loosely fill cupel mould and give piston a push 3 or 4 pretty hard blows with wooden hammer. — Remove cupel from

mould and dry. - It is best to allow it to dry at ordinary temperature, and not to use cupel until it is two or more months old. Still it can do no harm if it is dried at a higher temperature and thus prepared for immediate use. —

Charge for "Galena" (Lead Assay)

Ore - 10. grams
Blk. Flux Substitute - 35. grams.
3 - 5cm Nails. —
Salt Cover.

(Black Flux Substitute —
10. pts. B.C. Soda.
3. " Flour.

Iron Ore - Partial Analysis —

Determination of Phosphorous, Silica, Iron, and Sulphur.

Take 3. grams of finely pulverized ore, mix with 12 to 15 grams dry Na_2CO_3 + 3. grams KNO_3 or $NaNO_3$. Fuse in a platinum crucible. Dissolve fused mass when cold in water and HCl. Bring to dry mass on water bath, add HCl, + H_2O, digest + filter. Residue = SiO_2, which ignite and weigh. —

Iron. Of filtrate take an aliquot part, representing ½ gram ore, Note. Fe, Al &c. with NH_4OH — boil + filter + wash. Dissolve residue in H_2SO_4 (dil) divide into two equal parts, reduce with Zinc and titrate with standardized solution of Permanganate of Potass.

Sulphur. Take another aliquot part of filtrate from SiO_2, also representing ½ gram ore and precipitate SO_3 with $BaCl_2$. Boil, allow to settle, filter, dry, ignite & weigh as $BaSO_4$, & estimate as S. —

Phosphorous. Take balance of filtrate from SiO_2, representing 2. grams ore ppte. Fe, Al, P_2O_5 + etc. with NH_4OH, boil, filter wash. Dissolve ppte. in dil. HNO_3; filter if necessary. To solution add 50 to 100. cc. — solution Ammonium Molybdate (see p. 17.) heat to about 80°C or nearly to boiling and keep at that temperature for some time until precipitation is complete. Filter and wash thoroughly. Dissolve Molybdate ppte. on filter with dilute NH_4OH; and in solution ppte. P_2O_5 with Magnesia Mix-

turn; allow to stand over night, filter, wash with Ammonia water, dry, ignite + weigh as $Mg_2P_2O_7$ — + Estimate as P_2O_5 or P. —

(Combined C)

Carbon (~~Graphite~~) in Iron or Steels.

Color test with HNO_3. —

Take 0.1 gram of a sample of steel, which contains a known quantity of C. dissolve in 1.5 to 5. c.c. of HNO_3 (1.2 Sp. Gr.) then dilute it to a certain bulk so that each c.c. contains a known quantity of C.

Now dissolve a similar quantity of the sample to be tested in a similar quantity of HNO_3 (Sp. Gr. 1.2) when dissolved heat to 80° C. (no higher) on water bath, allow to cool, (this should also be done in making first solution). Place cold solution in graduated tube and dilute until it has the same color as ~~the~~ solution of steel with known quantity of C. — Read off bulk, and from this estimate

quantity of C. —

Solutions of Carmine or burnt coffee can be made of such a color as to represent solution of Am with known amount of C. These solutions will retain their color for an indefinite time, while the Am solution soon loses color. —

Pepsin. - Tests for the Determination of the Digestive Power of Pepsin.

Boil two fresh eggs for 15 minutes, cool and after freeing the coagulated albumen from all yolk and superficial moisture, press by means of a spatula, through a brass sieve having 20 to 30 meshes to the lineal inch. After thoroughly mixing the moist divided albumen weigh out 500 grains (33 grams) and transfer to a mortar. Measure out exactly 11 fluid ounces of water previously warmed to 100° F.; carefully triturate the albumen and part of the water, added in portions, and thereby obtain a uniform division of the particles, transfer this by the aid of the remaining water, to a flask having

a capacity of 16 fl. oz. or so; to this add 50 (fifty) minims of Hydrochloric acid, U.S.P. (s.g. 1.16, containing 31.9 o/o HCl.) and bring the fluid to exactly 104°F (40°C); then add 1/4 (one-fourth) grain (.017 gram) of Pepsin - noting the time of addition. Then immerse the flask in a water bath, heated to 104°F (40°C) exactly; after digesting six (6) hours, with frequent (every 15 minutes) rotation of the digesting fluid, remove the container and immerse it in cold water to stop digestion.

A comparison of the sample under examination, with a control flask, containing merely the albumin, acid and water will now determine the extent to which the albumin has been dissolved, and approxim-

ately the amount of Pepsin which would be needed to accomplish entire solution of the albumen. If necessary the test must now be repeated with addition of sufficient Pepsin to produce such a result and the quantity required will be a direct indication of the power of the sample.

The proportions given above are to be used with the pure (powder or scale) Pepsin. If Pepsin has been diluted sample taken should be accordingly larger.

Of Parke, Davis & Co. "Pepsinum Purum Pulvis (Pure Powdered Pepsin) 1. part dissolved 2000, parts coagulated White of Egg.

Of P. D. & Co's Pepsinum Purum in Lamellis (Pure Scale Pepsin) 1 pt. dissolved 2000, parts coag. White of Egg.

Of Boricki Pepsin – (1–5 Milk sugar) 1 pt. dissolved 1/53 parts coagulated white of Egg q.s. –

White of Egg contains about 11 to 13 % (?) Albumen. Parke, Davis & Co. say that White of Egg contains q.q. & practically 10 % of Albumen – proof

The above process works remarkably well.

$Mg_2P_2O_7 \times 0.2793 = P.$

$BaSO_4 \times 0.1373 = S.$

1 pt. slaked lime ($Ca H_2 O_2$) dissolves in 700. parts of cold water. or - 84. U.S. gallons of cold water will dissolve 1 lb. of $Ca H_2 O_2$ to a clear, saturated lime water.

Analysis of supposed sample of Steel (analysis really indicates Pig Iron) Oct. 1890. —

P.	0.5697 %
S.	0.0936 "
Si.	2.0290 "
C (Combined)	0.5791 "
Mn	0.2455 "

Nov. 10 — 1890 —

Analysis Steel (? Pig Iron)

P.	0.0566
S.	0.0824
Si	1.9290
C (Combined)	0.4032
Mn	0.3295

Steel analysis. (Or Pig Iron.)

Determination of P, S, Si, C (Combined) & Mn.

Phosphorus (P.) Sample = 5. grams. Dissolve in equal pts. HCl & HNO_3, 30. c.c. to each gram, in a beaker, adding acids all at once - to prevent escape of P. - Evaporate on Sand bath - nearly to dryness - then remove to air bath and heat at 130° to 140° C, until res. is quite dry - brittle - not sticky. Add 40. c.c. HNO_3 and heat on sand bath until Fe is dissolved. Evaporate about half the HNO_3. Add H_2O and filter & wash. Add 100. c.c. Molybdic solution - then a few c.c.s NH_4HO - so long as ppte. dissolves very readily. This to prevent HNO_3 being too strong. Heat to 70° or 80° C., stir well & let stand over night - Filter, test filtrate for P - wash with Molybdic sol. - Dissolve in NH_4HO and ppte. with Magnesia mixture. Weigh as $Mg_2P_2O_7$.

Sulphur (S.t Sample = .5 Grams. Place in flask supplied with funnel tube and connected with Wills bulbs containing HCl solution of Br. Add 50 cc. H2O to flask. Then add HCl to flask in small quantities at a time keeping up a brisk effervesence. H2S passes over with it and is oxydized by Br to H2SO4. When effervesence has nearly ceased add large Excess of HCl and heat. Disconnect—evaporate Br solution to small bulk. Filter contents of flask wash and dry residue. Digest with aqua Regia over night and heat to 50°C. for several hours, to oxidize balance of S. Dilute with water, filter—mix filtrate with Br solution and evaporate on sand bath. When evaporated to 50 cc. add BaCl2 & continue to evaporate on Sand and water baths, until nearly dry. Add two drops

HCl and water heat, allow to stand, filter, wash, ignite & weigh as $BaSO_4$. (N.S. instead of digesting residue with aqua regia, it may also be fused with Na_2CO_3 + KNO_3) —

Silicon (Si)

Residue from P det. can be used. Ignite res. & fuse with Na_2CO_3 (and KNO_3 if much graphite is present.) Dissolve in H_2O & HCl — Evaporate to dryness — etc., separating SiO_2 in usual way. Estimate as Si. —

If it is desired to use larger sample (5. or 10. grams) — place it in porcelain dish add water and gradually HCl. Heat & evaporate to dryness on sand bath & air bath at 130 to 150°C. Take up in HCl & water. Filter, dry, ignite and fuse and continue as above.

Combined Carbon (C) Sample = 0.1 gram. HNO_3 (1.2 Sp. gr.) 5. cc. (20. cc. conc. HNO_3 + 25. cc. H_2O = 1.2 Sp. gr.) See p. 140.

Manganese (Mn). Sample = 3. grs. Dissolve in Aqua Regia – evaporate to dryness to separate SiO_2. Dissolve in HCl + H_2O – filter + wash. Ppte. Fe as acetate as follows – add Na_2CO_3 to a deep red, then a few drops HCl – also acetic acid – excess of sol. Sodium acetate + boil. Dilute with boiling water to 1000. cc. Allow to settle or filter, after thoroughly mixing. Take aliquot part of clear liquid – say half. Heat to boiling (it is well to concentrate to about 300. cc.) add Na_2CO_3, boil and add a little Na hydroxide, to ppte. Fe left, Ca, Mn etc. Allow to settle, filter and wash. Dissolve ppt. in small quantity of HCl – adding a little sulphurous acid if it dissolves with difficulty. Boil filtrate, with few drops HNO_3. Add Na_2CO_3 until color deepens slightly – previously adding a little Fe_2Cl_6, if neces-

+ any as indicator. Next add Sodium acetate and boil & ppte. Fe. Filter and wash. - Ppte. Mn. in filtrate by heating with a few drops Br. Filter & wash, testing filtrate by adding a little more Br. and again heating. Dry and ignite ppte. and weigh as Mn_3O_4.

Graphite in Pig Iron, Sample 2 to 4 gms. Dissolve Fe in HCl - boiling. Filter and wash thoroughly. Boil residue with NaHO or KHO & dissolve SiO_2. Filter and again wash thoroughly. Again boil residue with HCl filter on same filter, wash dry and weigh as C. (Graphite)

Mn_3O_4 - 229.	— 100.	
Mn_3 — 165.	72,05	per cent.
$Mg_2P_2O_7$ - 222.	— 100. -	
P_2 — 62.	27.93	" "
$BaSO_4$ 233.	— 100. -	
S — 32.	13.73	" "

SiO_2	60.	100.—
Si	28.	46.67
O_2	32.	53.33

Analyses of some Samples of Dry Cal. acid Phosphate.—

	"Bread acid." St Louis.	"C. a. P." St Louis.	St Louis.	National Chem. Wks.
H_2O.	5.48	5.95	6.62	7.27
Starch.	33.43	41.36	41.27	34.59
$CaSO_4$.	1.58	1.14	1.16	2.44
$Ca\,H_4P_2O_8$	43.58	39.15	35.18	28.58
H_3PO_4	10.80	7.32	8.03	10.97
$Mg\,H_4P_2O_8$.			2.42	13.19
$Cu_2H_2P_2O_8$.	4.70	4.13	1.97	
~~Fe Sesquioxide~~.	Iron & Alumina, Ac. Phos.		.81	0.69
100. pts. neutralize of $NaHCO_3$	Water & undetermined, by Diff.		2.54	
	49.28	40.57		51.15

Analyzed a Sample of acid Phosphate (Dry) which contained some 30.% of $CaSO_4$ and was sold as Cream of Tartar.—

Analysis of Acid Phosphates and Mineral Compounds in which Phosphoric acid predominates.

Acid Phosphate.-

H_2O.- Dry sample at 110°. until weight is constant. Diff. = H_2O.-

General Course of Anal.-

Take sample of dry 4. grams- add about 100. c.c. water and allow to stand for some hours, stirring from time to time. Filter & wash- dilute to 200. c.c. (A.)

Wash res. with Dil. HNO_3 & then water & dissolve insoluble phosphate. ~~Weigh & Dry & weigh~~ res. as Starch. In filtrate ppte. P_2O_5 with Ammon. Molyb. and then with Magnes. Mix. in usual way.- Calculate as $Ca_2H_2P_2O_7$.-

P_2O_5. (Soluble) Of 200. c.c. (A.) take 10. c.c. = 0.2 gr. & det. P_2O_5 by double precipitation as usual.

Fe Al $_2O_3$. Of 200. cc. (A) take 50. cc. = 1. gram. Add NH_4HO in slight excess and then acetic acid in slight excess, filter and wash. Res. = Fe Al phosphates. Dry ignite and weigh. - ½ of weight approximately = Al_2O_3 + Fe_2O_3. -

CaO. To filtrate from Fe Al add a few drops of HCl and then an excess of Ammon. Oxalate. Boil, filter, wash, ignite ~~filter~~ ~~blast~~ blast lamp & weigh as CaO. —

MgO. To filtrate from CaO add NH_4HO in excess, allow to stand, filter, wash with dilute NH_4HO, filter, dry ignite & weigh as $Mg_2P_2O_7$.

NaHK. To aliquot portion of 200. cc. (A) add excess of $BaCl_2$ + NH_4HO + phosphate. all Ppt. - Filter & wash. To filtrate ppte. excess of $BaCl_2$ with $(NH_4)_2CO_3$ - filter, evaporate filtrate to dryness & ignite. If necessary dissolve Res. in H_2O + ppte. with $(NH_4)_2CO_3$ a second time,

Weigh Alkalies as Chlorides.

SO_3. Take aliquot portion of 200. cc. (A) add HCl & ppte. with $BaCl_2$ & weigh in usual manner.

Fe. If it is desired to det. Fe separately take aliquot portion of 200. cc. (A) add H_2SO_4 and filter off $CaSO_4$ if necessary. Dilute with H_2O - reduce with Zn and titrate with Potass. Permang.

Calculation. Combine insoluble P_2O_5 with CaO - Balance of CaO with Sol. P_2O_5 as $CaH_4P_2O_8$; MgO " " " " $MgH_4P_2O_8$ - Fe & Al " " " " $FeH_4P_2O_8$ & $AlH_4P_2O_8$. -

Na_2O ($+K_2O$) with P_2O_5 as NaH_2PO_4. SO_3 with CaO - before - omitting latter with P_2O_5; —

Analysis of "Phosphor-Bronze."

Copper —	80.647	% —
Lead —	7.619	"
Tin (Sn) —	10.212	"
Phosphorus (P)	1.332	"
	99.810	

Phosphorus. — Take a sample of about 1. gram of bronze, boil in porcelain dish with HNO_3 (S. G. about 1.3) and evaporate nearly to dryness. Dilute with water and filter. Most of the P is converted into Stannic Phosphate and is mixed with the Meta-stannic acid in the residue. (A) The filtrate may still contain traces of P, which ppte. with Ammon. Molybdate, filter, wash and dissolve in NH_4OH in usual way — Sol. B. —

Now wash Res. A. into a beaker by puncturing filter-paper in

funnel, while still wet, first with a little conc. Aqua Regia and then with water. Without waiting for Stannic Oxide & Phosphate to completely dissolve, add at once an excess of Ammonia and then of freshly prepared, colorless Ammonium Sulphide ($(NH_4)_2S$.) stir and allow to stand for an hour or two. Then filter, wash with $(NH_4)_2S$ and mix filtrate with filtrate "B." Precipitate the P in these mixed solutions with Magnesia Mixture, filter wash, ignite and weigh in usual way as $Mg_2P_2O_7$.

Process gives excellent results.

Sn, Pb & Cu. - Take sample of about one gram, boil with HNO_3 (Sp. 1.3) in Porc. dish and evaporate nearly to dryness. - Add water and filter and wash. Res. = Sn, P, and traces of Cu & Pb. - Filtrate = most of the Pb & Cu. -

Dry Res, ignite filter separately, introduce dried Res. into porc. Crucible with ash, add about 5 times the weight of a mixture of equal parts of Na_2CO_3 and pure Sulphur, and fuse for some time over Bunsen burner. Allow to cool, take up in water and filter. Wash with H_2S water. Filtrate (3) contains all the Sn. Res. contains traces of Cu + Pb Sulphides, which wash into the solution containing most of the Cu + Pb.

To filtrate "3" add a slight excess of HCl, heat for some time until odor of H_2S has disappeared filter, wash with water containing Ammon. Acetate and few drops of acetic acid, Res. = all the Sn and considerable Sulphur. Dry, remove from filter, which ignite in porcelain Crucible. Then

introduce the dry Cin Sulphide heat at first slowly to expel Sulphur, then to redness, and finally over blast lamp. - Weigh as SnO_2. —

Pb + Cu. Evaporate the solution (HNO_3 filtrate from Sn) now containing all the Cu + Pb in porcelain dish to a small bulk, add 10. c.c. Conc. H_2SO_4 and heat over bunsen burner on gauze, until dense white fumes of H_2SO_4 arise. allow to cool, dilute with water, filter and wash. -

Res. = all the Pb, which dry and ignite in usual way and weigh as $PbSO_4$. -

Filtrate = Cu. - Dilute to 200. c.c. - Of this take aliquot portion, place in Platinum dish and ppte Cu with Zinc; weigh as Cu. See Treasures — Page 314 —

Analysis of Spelter:-

Sn + Sb -	0,79 %-
Pb ———	0,98 "
Cu ———	0,14 "
Fe ———	0,23 "
Zn ———	98,36 "

Sn + Sb (+ SiO_2). - Take sample of about 5 grams, treat in porcelain dish with ~~aqua~~ HNO_3 of about 1.3 Sp. Gr. - Evaporate nearly to dryness, take up in water and filter. Res. = Sn + Sb. (+ Si) - Dry, ignite in usual way and weigh as SnO_2 + Sb_2O_4. Calculate to Sn + Sb. -

Pb. Evaporate filtrate from Sn + Sb to small bulk, add 10. c.c. H_2SO_4 (conc.) and heat on gauze over bunsen burner until dense white H_2SO_4 fumes arise. Cool - add water filter and wash. Res = $PbSO_4$, which

dry, ignite and weigh in usual manner.—

Cu. Take all or aliquot portion of filtrate from $PbSO_4$.— place in platinum dish and pte. Cu either electrically or ~~by~~ with Zinc.—

Fe. — Dissolve 5. grams of pelter in dil. H_2SO_4 and titrate with Potass. permanganate solution.

Zn — by difference.—

Analysis of alloy of Pb, Sn & Sb.—

Treat sample (say 1 gr.) in porcelain dish with HNO_3 (1.42 g.) evaporate to dryness on water bath. Take up in water, filter and wash. In filtrate determine Pb as $PbSO_4$ in usual manner.—

The residue on filter, containing Sn & Sb, is dried, then separated as completely as possible from filter, the filter incinerated, the whole of the Sn & Sb together with filter ash transferred to Silver crucible, moistened with few drops of NaHO sol. and dried.— Then add of dry pure NaHO about eight times the wt. of Sn & Sb. Heat gradually until fused and fuse for some time at red heat.— Allow to cool, dissolve in hot water, filter, wash residue first with mixture of equal pts. water & alcohol, then

with mixture of three pts. alcohol + 1. water.

Filtrate = Sn. –

Res. (A.) = Sb. –

To alcoholic filtrate add HCl & acid reaction – ppte. the Sn with H_2S, acid determine as SnO_2 as given on page 160. –

Sb. Dissolve Res. (A.) on filter-paper with mixture of equal parts of HCl and Tartaric acid. – Dilute with water & ppte. Sb. with H_2S. Determine as Sb oxide as given in Fresenius – p. 334. H

Analysis of Florida Mineral Phos-
phate. Determination of —
$Al_2O_3 + Fe_2O_3$, P_2O_5 ($Ca_3P_2O_8$), & H_2O, —
Nov. 4. - 1891. —

	H_2O.	Fe_2O_3 & Al_2O_3	P_2O_5	$Ca_3P_2O_8$
No. 1 —	2,76	2,60	37,18	81,16
" 8. —	1,55	2,61	36,19	79.01
" 14. —	0,58	1,18	36,66	80,03
" 5. —	0,95	0,94	38,43	83,89
" 6. —	1.39	1,81	38,46	83,95
" 9. —	2,23	2,76	35,58	77,67
" 20. —	1,38	1,39	37,28	81,38,
Average of 20 samples	1,54	1,66	36,81	80,37

Course of Analysis: —

H_2O. Heat sample to a dull redness, not hot enough to drive off ~~water~~ CO_2, Diff = H_2O, or ~~dry~~ at 100.°C and report as such. —

P_2O_5 ($Ca_3P_2O_8$). Take a sample

of 5. grams - dissolve in a mixture of 25. c.c. HNO_3 (1.2 Sp. Gr.) and 12,5 c.c. conc. HCl by boiling in a covered beaker for several minutes. Filter, wash and dilute filtrate to 250. c.c. Discard residue. Of the 250. c.c. filtrate take 10. c.c. = 0.2 gram sample, place in porcelain dish, add a few c.c. HNO_3 and evaporate on plate or gauze until all the HCl is expelled. Dilute with a little H-O, warm & beaker, add 100. c.c. Molybdate Solution and a few drops NH_4OH, heat, allow ppt. to subside, filter, wash, dissolve ppt. in NH_4OH, ppte. with magnesia mixture, filter, wash, dry & ignite through as $Mg_2P_2O_7$ in usual way.

Fe_2O_3 & Al_2O_3 - [Glaser's Method slightly modified.] -

Of the 250. c.c. filtrate take 50. c.c. = 1. Gr. add 10. c.c. of diluted

H_2SO_4 (1. Conc. H_2SO_4 to 4. of water by volume) - boil for some minutes until liquid is concentrated to 50 c.c. or even to 40 or 35. c.c. - Transfer to 200. c.c. flask, add alcohol (preferably freshly distilled) up to mark. Shake thoroughly, and allow to stand, with occasional shaking for 12. hr. so that the $CaSO_4$ may separate completely. Filter off just 100. c.c. (= 0.5 gr. sample), boil until all the alcohol is expelled, replenishing with H_2O. Now add slight excess of NH_4HO and again heat to expel excess of NH_4HO, thereby promoting the co-precipitation of Mg. Now filter and wash 3 or 4 times without disturbing ppte. on filter. - Dry, ignite and weigh. One-half the weight of the Phosphates of Iron and Alumina will be

the weight of $Al_2O_3 + Fe_2O_3$. — Report the two oxides together. Also calculate P_2O_5 to $Ca_3P_2O_8$ and report both ways. —

Hazen's Method for $Fe_2O_3 + Al_2O_3$ in Mineral Phosphates, is the one now universally adopted in Europe. (1892.)

Analysis of Zinc.

Determination of Sn, Sb, Pb, Cu, As, Cd, Hg (if any Amalgam) + Zn by difference + &c.

Take about 4. grams (or more) of Zinc Treat with HNO_3 (about 1.3 Sp. Gr.) in porcelain dish and Evaporate to Dryness as nearly as possible on water bath. Then moisten with a few drops of HNO_3, add water, digest and filter. - Res. = Sn + Sb, which may be ignited and weighed together. Or if it is desired to separate them proceed as ~~follows~~ on page 164. -

In the filtrate from Sn + Sb, considerably diluted, precipitate the Pb Cu As and Cd with H_2S. After adding some Acc. Filter and wash with H_2S water. - Res. = Pb, Cu, As + Cd. Dissolve Cd on moist filter paper by pouring over it repeatedly warm,

dilute H_2SO_4 ($1H_2SO_4$ to $5H_2O$).
Wash.—
Res. = Pb, Cu + As.—
Filtrate = Cd.—
In filtrate ppte. Cd by diluting largely and passing H_2S into it. Filter on Tared (counterpaised) filter, wash, dry and weigh.—
As. Dissolve as from Res. of Pb, Cu + As, by repeatedly pouring over moist filter, small amount of NH_4HS (freshly prepared is to be preferred). Wash with H_2S water. ppte. as in filtrate with Sol. H_2SO_4 in excess.— Heat moderately to expel H_2S + filter.— Dissolve as in Aqua Regia on filter paper heat for a time, add excess of Tartaric acid, then excess NH_4HO and Magnesia Mixture; allow to stand 12 hours, filter, wash with NH_4HO water, dry, ignite and weigh as $Mg_2As_2O_7$.—
Acidify filtrate from As with Ac

and test for Pb with H_2S.—
Pb. The PbS + Cu S still in filter and still moist, is dissolved in boiling dil. HNO_3. Evaporate nearly to dryness in porcelain dish, add 6. to 10. cc. Conc. H_2SO_4 and heat on gauze or sand bath until dense fumes of H_2SO_4 escape.— Cool, dilute with some water add alcohol, allow to settle and filter off $PbSO_4$, which weigh in usual way.—
Cu. Heat filtrate to expel alcohol, place in Pt. dish and ppte. Cu with Zn or electrolytically.—
Fe. Dissolve 1. or more grams of sample in dil. H_2SO_4 and titrate with Permang. of Pot. solution.
Hg. (Hamy). Dissolve sample in $HCl + HNO_3$, evaporate nearly to dryness on water bath — until acid fumes have nearly been dispelled. Take up in water with a little HCl — filter and wash.

Dilute somewhat and ppte Hg as Hg_2Cl_2 by adding excess of either phosphorous or hypophosphorous acid. Allow to stand for 1½ hours; filter and wash on Tared (counterpoised) filter, Dry and weigh as Hg_2Cl_2.

Analysis of sample of Battery Zinc - Nov. 18 - 1892.

Pb	0.918	%
Cd	0.272	"
Fe	0.131	"
Sb	0.012	" (Very low)
Cu	0.035	" (low)
Cl	trace	"
Zn (by diff)	98.632	"
Sn	none or trace.	

Free & Albuminoid Ammonia in Potable Water. —

1891.

According to Wanklyn a good Potable water should not contain to exceed 0.05 parts of Free Ammonia and 0.10 parts Albuminoid Ammonia per Million of water.

Determination of Glycerine in Soapboilers. (Bi-Chromate Process)—

1,00 Gl. glycerine requires for oxidation 7,486 pts. Potass. Bi-Chromate.

Standard Solutions:—

1. Potass. Bi-chromate sol. containing 80. grammes Bi-chromate and 150. cc. Conc. N-SO4 per Litre.— The Exact value may be determined by standardizing with Iron wire or Ammon. Ferrous Sulphate.

2.— Ammonio Ferrous Sulphate sol. containing 120. grams per Litre as well as some N-SO4—

3. Bi-chromate ten times as dilute as above.—

The Ferrous sol. is standardized to Bi-Chromate sol.—

Process:— Take of the Lye about 30. grams. add to it an excess of sol. Basic acetate of Lead and dilute to 750. cc

Shake thoroughly & allow to settle.-

Take of this 50.cc.- filter, add H_2SO_4 & pot. excess of Pb.- Filter again.- Dilute to about 200 or 250.cc.- add 25.cc. Conc. H_2SO_4 (diluted) and then 50.cc.- Standard Bi-chromate sol.- Heat to near boiling for 2 hours. then add excess of Ammonio-Ferrous sol. and titrate that back with Bi-Chromate sol., using Ferricyanide as indicator. It is well to make the final titration with the Deci-normal Bi-Chromate sol.-

Calculate from the chromate consumed the amount of Glycerine. —

Analysis

Illinois Coals made by

Location of Mine	Heating Power BTU
La Salle. --- La Salle Co	12169
Peru. ---- " " "	11829
Bloomington. -- Mc Lean Co	11313
Oakwood. Vermillion Co	12528
Fairmont " " "	11431
Danville " " "	12285
" " " "	12506
" " " "	11655
Lincoln Logan Co	11275
" " "	11718
Mt Pulaski " "	11360
Niantic Macon Co	11529
" " "	11529
Riverton	11633
"	12096
Barclay	11205
Pana. Christian Co	11610
Assumption " "	13608
Mount Olive	11441
" "	11772

Of

The University of Illinois 1891

Vol % Matter	Fixed % Carbon	ash %	Moisture %
39.40	43.95	8.43	8.22
37.19	47.20	6.61	9.00
35.99	45.20	14.71	4.10
37.55	46.00	8.65	7.80
31.81	47.05	13.05	8.10
32.35	53.00	3.65	11.00
43.70	45.37	6.15	4.78
37.03	46.43	10.88	5.62
34.99	44.50	12.06	8.45
31.30	49.50	11.70	7.50
35.82	46.53	9.97	7.68
36.25	47.40	8.47	7.88
34.15	45.50	8.50	11.85
35.29	48.45	9.78	6.38
38.45	44.30	6.15	11.10
35.67	46.25	10.71	7.37
36.37	46.95	9.46	7.22
41.20	50.10	4.80	3.90
35.43	44.49	11.11	7.97
35.00	51.50	8.20	5.30

Analysis

Illinois Coals made by

Location of Mine		Heating Power B.T.U
Odin Marion Co		11954
" " "		12663
" "		11664
Sandoral " "		11959
Centralia " "		11781
DuQuoin Perry Co		12170
" " " "		12393
Big Muddy Franklin Co		12825
Carbondale Jackson Co		12582
Am. Stoker Co #47	Fr 1.29 S 2.50	13821.2
" " " 48	Fr 1.41 S 2.88	12368.3
Ch & Ind Co	S. 1.96	14189.36
" " " "	S. 1.13	13845.2
Hasler - Coffee	S. 1.38 P. 0.061	
Linden Fuel	S. 4.22	10225.
Ch. & Ind Co "Ills"	S. 2.296	13821.15
Linden Fuel Co	~~S. 4.37~~ ~~Fr 3.78~~	11330.61
" " "	S. 4.09 Fr 3.74	12027. 12078.72 E
Selby Town Ill.		13387 - E
Linden Fuel	S. 1.08 Fr 1.28	12069.75

Of Con. on Page 191

The University of Illinois 1894

Vol. Matter %	Fixed Carbon %	Ash %	Moisture %
33.94	50.86	9.08	6.07
41.10	46.90	6.10	5.90
34.00	47.45	11.55	7.00
35.07	50.86	6.88	7.19
38.18	45.50	8.04	8.28
32.03	53.69	7.44	6.84
30.35	54.08	8.90	6.67
32.15	56.00	4.00	7.85
33.65	57.50	4.60	4.25
38.23	48.53	7.40	4.55
36.98	43.16	12.13	6.32
48.57	44.22	3.81	3.40
39.21	51.22	3.71	5.86
2.16	83.21	14.40	0.23
37.81	34.11	19.68	8.40
45.69	44.54	8.27	4.50
41.42	40.82	12.09	5.62
37.29	41.34	16.82	4.65
42.08	47.25	6.10	4.60
35.20	51.28	8.86	4.68

Milk Analysis

Fat. 25 cc or more milk + same quantity N Cl. C.P. Con mixed and heated to a red-brown color. Cool + shake well with 3 portions of ether in Separating funnel. Evaporate ether in a weighed bottle + weigh. Dif = the fat.

Ash 25 cc milk + 10 cc HNO_3 Con in Pt dish boiled dry with care, ignited + weighed. Dif = ash

Formalin in Milk

100 c.c. of sample heated to 70°C with 4 to 5 drops of H_2SO_4, dry powdered Na_2SO_4 added. Distill the first 50 c.c. and add Reagent bleached with $NaHSO_3$. Turns red or violet with HCl. (2) Turbid with sulfuric acid

Formaline.

(3) yellow ppt with Nessler's reagent, (4) turbid with phenylhydrazine + NaOH, blue with dimethyl analin.

(Analyst '99 pg 151)

Uchinn Test.

Dilute milk with an equal volume of water. A little 91% H_2SO_4 run in in a layer at the bottom (1 drop of Fe Cl). In the presence of Formaline, a violet blue color appears at the junction of the two liquids. The color is permanent for 2 or 3 days. Sensitive to 1/in 200,000.

Milk in absence of Formaline gives but a greenish tint + later a brown. Chromatin is but not in large amounts.

H. D. Richardson
Dairy Chem 1899

Boracic Acid in Meat

5–10 grms meat. ignite + fuse with K_2CO_3. dissolve in water and filter. Filtrate contains Boracic acid. Neutralize with HCl, add Magnesia mixture + NH_4Cl + excess to dry mass in Pt. dish + ignite. Treat with boiling water, filter and ppt with $AgNO_3$. Ignite MgO + MgBO strongly + weigh. Dissolve in HCl ppt MgO with Sod. phosphate in usual way. Calculate B_2O_3 by difference deducting MgO from mixed MgO + MgBO

Formula for Solids in Milk

Let S = Sp. G.
" f = % Fat
" x = Solids not fat

$$\left(\frac{100\,S - 5f}{100 - 1.0753\,5f} - 1\right) \times (100 - f)\,2.6 = x$$

Silicon in Steel

100 c.c. HNO_3 Sp.G 1.20
125 cc. H_2SO_4 Sp.G 1.28 mixed
5 grms turnings and 30 c.c.
added carefully in a Pt dish
covered with watch
glass. Bring to dry mass
until white fumes come
off. add 30 cc HCl. water
heat until all iron is dis-
solved, filter, wash, burn &
weigh in Pt crucible.
Add 1 drop H_2SO_4 Con & 5
to 10 cc H.F. evaporate carefully
burn & weigh. Difference in
wt equals Si

Manganese in Steel

HNO_3 1.14 Sp.G. (1 to 3 water) or 1.16 Sp.G. (900 to 1500)

H_2O_2 50 cc to 1000 water + 2.5 cc H_2SO_4 (1.04 Sp.G. to above.

$K_2Mn_2O_8$. 1.75 gms to 1000 water. titer with iron and make 1 cc = to .1% Mn when 1.5 gms are taken

Use 1.5 gms turnings and 30 cc above acid in large beaker glass. Boil until all steel is dissolved and the solution is syrupy add 40 cc HNO_3 conc acid and boil to dissolve all iron + Mn. Add $KClO_3$ in small quantities a until no Cl vapors are given off remove and cool beaker in cold water. Dilute to about 300 c.c. add 25 or more H_2O_2 solution from above, + titrate with standard titration or Iron

Manganese

quantity of H_2O_2 in 300 H_2O + 10 cc H_2SO_4 dil. Deduct this from the no. of cc used in the Mn titration. Difference in cc = % of Mn

---

Assay for Platinum.

Make 2 assays regular way on cupellation to the one for Pt add 10-12 parts of Silver c.p. same as for Au. Part the Pt bead with H_2SO_4 con. twice & weigh as usual. Treat the other with HNO_3 and weigh as usual

Weight H_2SO_4 bead = Au + Pt

" HNO_3 " = Au.

Difference in wt = Pt.

Gold = $20.67 per oz Troy
" " 4.30 ¢ " Gr.
" " .67 Mills p. Mgm
" " $1.04 per Gmt.

" "

1 grain = 16 pearls
1 Gr or 16 pls = $ 8/10 Tr Grains
1 Karat = 4 grains
15½ Karat = 1 Troy Oz
4 grs (Diamond) = 3 3/10 Troy Grs

Gold per ton $ 602,874.86

Gold = $20.67 per oz Troy
" = 4.3 ¢ " Grains
" = .66 ¢ " Grs
" = $1.03 " Gmt.

Grains per ton x by
.03215 = oz per ton

Analysis of

Coal.	Water %	Vol. Combustible %	Fixed Carbon %	Ash %
1.	3.32	34.81	41.99	19.88
2	4.55	38.23	48.56	8.69
3	6.32	36.98	43.16	13.54
4	44.50	45.69	44.54	5.27
5	2.40	48.57	44.22	3.81
6	5.86	39.21	51.22	3.71
7	8.40	37.91	34.11	19.66
8	6.62	41.12	40.87	12.39
9	4.05	37.29	41.84	16.82
10	4.60	42.05	47.15	6.20
11	4.68	35.20	51.26	8.86
12	6.79	40.57	30.62	22.02
13	10.66	41.69	43.30	4.45
14	7.40	39.65	39.83	13.17
15	6.60	36.85	38.10	18.45
16	7.48	34.79	36.35	21.38
17	6.20	34.87	45.43	13.60
18	5.97	39.79	31.76	20.67
19	11.07	31.28	40.25	17.00
20	0.45	21.35	66.88	11.35
21	4.30	40.65	29.45	25.60

Iron %	Sulphur %	Coke %	B.T.U.	
—	2.22	61.87	—	
3.92	2.50	—	13321	
10.44	1.88	—	12368	
—	2.296	49.81	13871	
—	1.961	48.16	14189	
—	1.127	54.98	13845	
—	422	63.79	10225	
—	—	53.26	11330	
3.78	4.39	58.66	12078	
1.64	2.64	53.36	15228	
1.26	1.08	69.12	12069	
3.88	6.75	52.64	11079	
—	2.26	47.76	—	
2.26	4.17	52.95	12272	
3.76	3.53	56.65	12754	
6.70	8.24	57.73	11072	
2.90	5.48	67.93	12,391	
4.40	3.57	63.43	11228	
2.93	4.16	57.65	11128	
2.46	2.18	78.70	13889	
4.52	7.33	55.06	10972	

Coal.	Water %	Vol. Combust. %	Fixed Carbon %	Ash %
22	6.08	37.92	47.16	19.84
23	5.80	38.20	50.75	5.25
24	10.46	32.44	36.05	21.05
25	10.27	34.20	36.53	19.00
26	1.50	21.12	64.29	13.09
27	11.20	44.28	43.96	1.56
28	7.60	35.96	36.70	19.74
29	6.31	19.54	58.53	15.62
30.	9.70	43.71	36.59	10.00
Coke.				
1	0.23	2.16	83.21	14.40
2	2.00	5.00	79.55	15.45

Iron %	Sulphur %	Coke %	B.T.U.	
2.79	4.36	56.44	11291	
0.86	1.24	57.00	13077	
1.09	1.42	56.00	13637	
3.35	4.33	57.10	10690	
3.57	2.13	55.53	11014	
2.00	2.69	77.38	13127	
0.45	0.61	46.53	13392	
4.94	2.93	74.15	10267	
2.20	2.12	46.59	12391.2	Freeman 6-18-03
—	1.38	—	—	Phosphorus .068
3.35	1.16	—	10275	.1

Marl.

Silica and Insoluble	2.04
Iron and Aluminum Oxide	0.72
Calcium Carbonate	88.22
Magnesium Carbonate	1.48
Water and Organic Matter	7.54
	100.00

Fullers Earth.

Silica (SiO_2) + Insoluble	72.190
Aluminum (Al_2O_3)	12.010
Iron Oxide (Fe_2O_3)	4.470
Lime (CaO)	1.370
Magnesium (MgO)	4.856
Water and Undetermined	4.404
	100.000

Fire Clay.

	1.	2.
Silica	84.66	85.86
Iron + Alumina	11.45	10.60
Lime	Trace	Trace
Magnesia	0.12	Trace
Loss on Ignation	3.99	3.51
	100.22	99.97

Sept. 18, 1906

Formula for extraction of Fluid Extracts and drugs.

10 gms. Fluid extract or dry drug.

If Fl. Ex. is used, mix with a sufficient quantity of boh saw dust on watch glass and evaporate over H_2SO_4.

Extract dry mass with mixture 25 cc $CHCl_3$ + 75 cc (80 + 15) after making alkaline with NH_4OH

Decant 75 cc = (7.5 gms)

Now shake with 1st 25 cc, then 15 cc, then 10 cc of H_2SO_4 (1% solution)

Make alkaline and shake with

20 - 10 + 10 cc CH_3Cl.

Evaporate in suitable glasses and examine the alkaloidal contents

---

Joe Williams Flux

Borax	140. - 280
B.C. Soda	180. - 360
Pearl ash	190. - 380
Pb O	230. - 460
Flour	40. - 80
	780 - 1560

Mix well

For heavy sulphide use two iron nails.

For heavy Copper use 3 nails

Cover 15 grs Borax

Formula for converting B° into Sp. gr. and conversely - Sp. gr. into B.°.

For liquids lighter than water :.

1. $\frac{144}{B^{\circ}+134} = Sp.\,Gr. \left\{ \frac{144}{Sp.\,gr.} - 134 = B.^{\circ} \right.$

2. $\frac{140}{B^{\circ}+130} = Sp.\,gr. \left\{ \frac{140}{Sp.\,gr.} - 130 = B.^{\circ} \right.$

For liquids heavier than water:

1. $\frac{144}{144-B^{\circ}} = Sp.\,gr. \left\{ 144 - \frac{144}{Sp.\,gr.} = B.^{\circ} \right.$

2. $\frac{145}{145-B^{\circ}} = Sp.\,gr. \left\{ 145 - \frac{145}{Sp.\,gr.} = B.^{\circ} \right.$

Of a 2½ % solution of Potas. chlor. —

1 drop. per quart = 1/10 grain per Gal. —
= 1,43 lbs. per 100,000. gals. —
or practically 15, lbs. to 1,000,000. gals.

1. U.S. Gal. = 58,328. grains.

To convert grains per imperial gal. into grains per U. S. Gallon, multiply by 0.83326 or 0.8⅓

Antimony in Lead 81.
Ash in Coal — 87.
Arsenic in Wall Paper 97.
Albumen in Beef Peptonoids 102.
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